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Fakulta textilní

Hodnocení hydrostatické odolnosti a komfortních vlastností prodyšných laminovaných textilií

Disertační práce

Studijní program: P3106 – Textile Engineering
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TECHNICAL UNIVERSITY OF LIBEREC
Faculty of Textile Engineering

Evaluation of Hydrostatic Resistance and Comfort Properties of Breathable Laminated Fabrics

Dissertation

Study programme: P3106 – Textile Engineering
Study branch: 3106V015 – Textile Technics and Materials Engineering

Author: **Abdur Razzaque, M.Sc.**
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Abdur Razzaque
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ABSTRACT

Waterproof breathable laminated fabrics allow water vapour passing through, but prevent liquid water to pass. This ability of the fabrics to protect rain and snow water while allowing sweat vapour to evaporate from inside to outside atmosphere, leads them to be used as outdoor sportswear or protective clothing. The challenge of enhanced hydrostatic resistance of these fabrics with proper breathability and other comfort properties has widened the research scope.

In the first part of the thesis, waterproof breathable laminated fabrics were prepared which could be used as outdoor sports fabrics. For this purpose, firstly, 20 GSM (g/m^2) microporous polyurethane (PU) membranes were laminated with three different types of polyester plain woven fabrics whose fabric weights and cover factors were different. Here, three different types of two-layered fabrics were prepared where polyester woven fabrics acted as outer layers and membranes acted as inner layers. And secondly, polyurethane membranes were laminated with those three different types of polyester plain woven fabrics and a one type of single jersey polyester knitted fabric to prepare three-layered fabrics. Here, three different types of three-layered fabrics were prepared where polyester woven fabrics acted as outer layers, membranes acted as middle layers and knitted fabrics acted as inner layers. All the newly produced six layered sample fabrics were characterized. Then this work investigated the influence of different parameters of these laminated fabrics, i.e., fabric weight, fabric thickness, fabric density, layered structures, warp and weft cover factors of outer woven layers on their different properties, i.e., hydrostatic resistance and other thermal and mechanical comfort properties, i.e., water vapour permeability, thermal resistance, air permeability, breaking strength and stiffness. It has been found from the test results and statistical analysis that there are significant influences of different fabric parameters on their different properties and these findings are important for designing and preparing waterproof breathable laminated fabrics for using as outdoor sports fabrics.

In the second part of the research work, a novel approach was applied for coating four different types of microporous polytetrafluoroethylene (PTFE) membrane laminated three-layered waterproof breathable fabrics. Here, eco-friendly C_6-based fluorocarbon water-repellent chemical and silicone based polysiloxane hydrophobic softening agent were used to prepare the coating solution in order to enhance the existing hydrostatic resistance and mechanical performance of the fabrics while keeping their breathability and air permeability in mind. From different test results and statistical analysis, it has been found that hydrostatic resistance and breaking strength have been significantly increased after coating while there are no significant changes in the results of water vapour permeability and air permeability. Furthermore, all the coated fabrics show good water-repellent property which is important for outdoor sports fabrics.

Keywords: Waterproof fabrics; breathability; hydrostatic resistance; water vapour permeability; water-repellent; thermal resistance; breaking strength; stiffness;

ABSTRAKT

Nepromokavé prodyšné vrstvené textilie umožňují průchod vodní páry, ale zabraňují průchodu kapalné vody. Tato schopnost textilií chránit před dešťovou a sněhovou vodou a současně umožnit, aby se pára odpařovala zevnitř ven, vede k použití pro venkovní sportovní oblečení nebo ochranné oděvy. Cílem práce je rozšíření klasifikace tkanin se zvýšenou hydrostatickou odolností s vhodnou prodyšností a dalšími komfortními vlastnostmi.

V první části práce byly připraveny nové vodotěsné prodyšné vrstvené tkaniny, které by mohly být použity na venkovní sportovní oděvy. Za tímto účelem bylo nejprve laminováno 20 mikroporézních polyurethanových (PU) membrán GSM (g/m^2) se třemi různými typy polyesterových hladkých tkanin, jejichž hmotnost a krycí faktory byly odlišné. Byly připraveny tři různé typy dvouvrstvých tkanin, kde polyesterové tkaniny působily jako vnější vrstvy a membrány působily jako vnitřní vrstvy. A za druhé, polyurethanové membrány byly laminovány těmito třemi různými typy polyesterových hladkých tkanin a jedním typem zátažného jednolícního polyesterového pleteného materiálu pro výrobu třívrstvých tkanin. Byly připraveny tři různé typy třívrstvých tkanin, kde polyesterové tkaniny působily jako vnější vrstvy, membrány působily jako střední vrstvy a pletené látky působily jako vnitřní vrstvy. Byly charakterizovány všechny nově vyrobené vrstvené textilie. Tato práce zkoumala vliv různých parametrů těchto laminovaných textilií, tj. hmotnosti, tloušťky, plošné hmotnosti, krycího faktoru atd., na jejich různé vlastnosti, tj. hydrostatickou odolnost a jiné tepelné, mechanické a komfortní vlastnosti, tj. propustnost pro vodní páru, propustnost vzduchu, pevnost v tahu a tuhost. Z výsledků testů a statistických analýz bylo zjištěno, že existují významné vlivy různých parametrů tkaniny na některé vlastnosti a tyto nálezy jsou důležité pro navrhování a přípravu nepromokavých prodyšných laminovaných textilií určených k použití pro sportovní oděvy.

V druhé části práce byl aplikován nový přístup s použitím potahovacího roztoku připraveného ekologicky optimalizovaným hydrofobním změkčovadlem na bázi fluoruhlovodíkové hydrofobní chemikálie a na bázi silikonu pro povlékání čtyř různých typů mikroporézních polytetrafluorethylenových (PTFE) vrstvených nepromokavých prodyšných sportovních tkanin pomocí metody suché vulkanizace, aby se zvýšila stávající hydrostatická odolnost a mechanická účinnost těchto tkanin při zachování jejich prodyšnosti a vzduchové propustnosti. Z různých výsledků testů a statistické analýzy bylo zjištěno, že hydrostatickou odolnost a pevnost v tahu se po povrstvení výrazně zvýšily, zatímco nedošlo k významným změnám ve výsledcích propustnosti vodní páry a propustnosti pro vzduch. Navíc všechny textilie vykazují správnou vodoodpudivou vlastnost, která je důležitá pro venkovní sportovní oblečení.

Klíčová slova: Voděodolné textilie; prodyšnost; hydrostatický odpor; propustnost vodních par; vodoodpudivost; teplotní odolnost; pevnost v tahu; tuhost;

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LIST OF ABBREVIATIONS

AATCC	American Association of Textile Chemists and Colorists
ANOVA	Analysis of variance
APEO	Alkylphenol ethoxylate
ASTM	American Society for Testing and Materials
BS	British Standard
GSM	Grams per square meter
ISO	International Organization for Standardization
LL	Lower limit
PFHA	Perfluorohexanoic acid
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctanesulfonate
PTFE	Polytetrafluoroethylene
PU	Polyurethane
R_{et}	Evaporative resistance
RWVP	Relative water vapour permeability
SD	Standard deviation
SEM	Scanning Electron Microscope
UL	Upper limit

CHAPTER 1: INTRODUCTION

This chapter briefly describes the background of the thesis followed by the objectives and overview of the research work.

1.1 Background

Breathability and waterproofing are two contradictory properties of fabrics. Breathability of a fabric is the ability of clothing to allow moisture vapour transmission from inside to outside atmosphere by diffusion and therefore facilitate evaporative cooling [1, 2]. It is determined by water vapour permeability [3]. On the other hand, waterproofing is a term that can be described as the impermeability to water. It is measured by hydrostatic resistance. Waterproof fabrics are resistant to the penetration of water even under pressure. These fabrics have fewer open pores and are less permeable to the passage of air and water vapour [4]. The resistance of a fabric to water depends on the nature of the fiber surface and the dynamic force behind the impacting water spray [5].

Waterproof breathable fabric is one of the harsh weather fabrics. In severe environmental conditions, waterproof breathable fabrics should protect the human body from external heat, wind and water, but at the same time they should let moisture vapour to be transmitted from inside to outside. These properties of the fabrics help to provide the wearer with a great level of comfort in many unpleasant situations. As a result, there are significant uses of waterproof breathable fabrics in the fields of sportswear and protective clothing [6, 7]. Waterproof breathable fabrics are of different types, i.e., closely woven fabrics, hydrophilic membranes and coating, microporous membranes and coating, combination of hydrophilic and microporous membranes and coating, smart breathable fabrics, retroreflective microbeads, and fabrics based on biomimetics [8-12]. Multi-layered waterproof breathable textile-polymeric fabrics are produced from various types of water-tight and wind-tight polymeric membranes that are permeable to water vapour. These membranes are of two basic types, like, microporous membranes that are mostly of hydrophobic character and hydrophilic membranes with compact structure [13-16]. Laminated microporous fabrics have holes that are much smaller than the size of the smallest raindrop, yet are much larger than the size of a water vapour molecule [17]. As a result, water droplets cannot penetrate the fabric, but water vapour molecules can penetrate. On the other hand, water-repellent fabrics resist being wetted by water. In this case, water drops roll off the fabrics [5]. By depositing hydrophobic materials on the fiber's surface, this type of fabric can be prepared. They have open pores and are permeable to air and water vapour. They still increase the water resistance property, but permit the passage of liquid water under higher hydrostatic pressure [4].

Again, comfort is a basic requirement during selection of clothing [18] and it is an integral part of the human body. It is one of the most essential attributes that a fabric should possess [19]. Comfort is associated with the ability of the body to maintain a constant core temperature under different environmental conditions, like cold or hot weather as well as under different physical or sports activities. Core temperature of the human body is approximately 37°C. When core body temperature exceeds 37°C under different conditions, perspiration is produced in order to balance the core temperature by secretion of sweat. It is important for clothing to play a role for keeping the body comfortable by removing sweat as water vapour [20]. So, water vapour permeability of a fabric which allows transmission of moisture and heat from the surface of human body into the environment is one of the most important factors. Because clothing comfort sensation is determined mainly by a balanced process of moisture and heat exchange between human body and environment through clothing system [21]. Sports apparels should prevent excessive heat loss in cold weather and enable the release of sweat from the surface of skin in hot weather. Three main categories of clothing comfort are tactile comfort, thermal comfort and aesthetic comfort [22]. Thermal property, air permeability, water vapour permeability and liquid water permeability have been suggested as critical properties for thermal comfort of the clothed body [23]. Besides, bending rigidity and breaking strength are considered as mechanical comfort for the wearers.

This research study firstly designed and prepared microporous polyurethane (PU) membrane laminated waterproof breathable fabrics and focused on assessing the effect of their different fabric characteristic parameters on their different properties, like, hydrostatic resistance along with their thermal and mechanical comfort properties which play vital roles for the users of outdoor sports fabrics. Secondly, this study applied a novel approach for coating four different types of microporous polytetrafluoroethylene (PTFE) membrane laminated three-layered waterproof breathable fabrics in order to enhance their existing hydrostatic resistance and mechanical performance.

1.2 Objectives

The goal of the research work was to prepare waterproof breathable membrane laminated fabrics and to evaluate their hydrostatic resistance and comfort properties with statistical analysis as well as to statistically analyze the enhancement of hydrostatic resistance and mechanical performance of multi-layered breathable waterproof fabrics. The specific objectives are as follows:

To prepare waterproof breathable fabrics

The objective of this work was to prepare and develop waterproof breathable membrane laminated fabrics which could be used as outdoor sports fabrics. Firstly, two-layered waterproof breathable fabrics were prepared using polyurethane (PU) membrane and three different types of polyester plain woven fabrics and secondly, three-layered waterproof breathable fabrics were prepared with those three different types of polyester plain woven fabrics, PU membrane and a one type of single jersey polyester knitted fabric.

Analysis of hydrostatic resistance of the prepared samples

The objective of this study was to determine the influence of different structural characteristics of the prepared PU membrane laminated two-layered and three-layered fabrics on their water resistance property which is very important for outdoor sports fabrics. Statistical analysis along with ANOVA (analysis of variance) was used to analyze the significance of various factors of the fabrics on their hydrostatic resistance property.

Study of thermo-physiological and mechanical behaviors of the prepared samples

This study was performed to determine the influence of different characteristics of the prepared PU membrane laminated fabrics on their thermo-physiological and mechanical behaviors. The thermal behavior was evaluated with the help of thermal resistance evaluation. Water vapour permeability was measured and analyzed in order to study the breathability performance of the sample fabrics as well as air permeability was evaluated. Mechanical behavior, i.e., breaking strength was identified with the result analysis of the breaking force of the fabrics and bending rigidity of the sample fabrics was analyzed. Statistical evaluation with ANOVA was used to analyze the significance of various factors on desired properties.

Application of a novel approach for coating

A novel approach for coating was applied on four different types of polytetrafluoroethylene (PTFE) membrane laminated three-layered waterproof breathable fabrics using eco-friendly C_6-based fluorocarbon water-repellent chemical and silicone based polysiloxane hydrophobic softening agent in order to enhance their existing hydrostatic resistance and mechanical performance while considering their water vapour permeability and air permeability. Water-repellent property of these four sample fabrics was also analyzed after coating. Statistical analysis of variance was used to describe the significant or non-significant increases of different properties of these four different three-layered fabrics after coating.

1.3 Overview of the research work

Waterproof breathable fabrics used for various protective clothing and sportswear should show the resistance to the penetration of water under pressure, while simultaneously should show a high possible permeability for water vapour. This permeability is of importance for the fabric's hygienic value, as it allows human perspiration to be carried away from the skin surface and secreted beyond the area between the clothing and the human body. This prevents the human body from overcooling or overheating and consequently protects them against a chill and ensures a good frame of mind and comfort [13-16, 24-29]. It is a big challenge to maintain hydrostatic resistance value and simultaneously water vapour permeability in the fabric. However, waterproof breathable fabrics had already been studied by some researchers. Kang et al. worked on waterproof breathable fabric prepared by electrospun polyurethane (PU) nanoweb to polyester and nylon blended fabric and discussed about its air and water vapour permeability with water resistance [6]. Ahn et al. studied on two-layered fabric laminated by electrospun PU nanoweb with water repellent nylon fabric and discussed about its waterproof and breathable properties [17]. Kim et al. explained thermal comfort and waterproof breathable properties for polyester fabrics with aluminum coated polyurethane nanoweb [30]. Ozen tried to develop waterproof breathable fabrics using plain and twill woven fabrics of cotton and polyester yarns and it was revealed from the study that waterproofing was increased when water repellent fabrics were laminated with breathable linear low density polyethylene films [4].

But, this research study newly designed and prepared microporous polyurethane (PU) membrane laminated waterproof breathable fabrics that could be used as outdoor sports fabrics. For this purpose, firstly, polyurethane membranes were laminated with three different types of polyester plain woven fabrics whose fabric weights as well as warp and weft cover factors were different in order to produce two-layered fabrics. Here, three different types of two-layered fabrics were prepared. Polyester woven fabrics acted as outer layers and membranes acted as inner layers. And secondly, polyurethane membranes were laminated with those three different types of polyester plain woven fabrics and a one type of single jersey polyester knitted fabric to prepare three-layered fabrics. Here, three different types of three-layered fabrics were prepared. Polyurethane membrane acted as a middle layer and knitted fabric acted as an inner layer for each of three different types of three-layered fabrics. But, outer layers of these three different laminated three-layered sample fabrics were three different types of polyester woven fabrics. Then all the newly produced layered six sample fabrics were characterized and evaluated statistically in order to analyze the significant influences of different fabric characteristic parameters on their different properties, like, hydrostatic resistance, thermo-physiological behaviors, i.e., thermal property with air and water vapour permeability as well as mechanical properties, i.e., bending rigidity and breaking strength.

In the second part of the research work, a novel approach was applied for coating four different types of microporous polytetrafluoroethylene (PTFE) membrane laminated three-layered waterproof breathable fabrics. For this purpose, water-repellent coating solution was prepared by ecologically optimized C_6-based fluorocarbon chemical instead of conventional C_8-based fluorocarbon chemical. C_6-based fluorocarbon chemical produces perfluorohexanoic acid (PFHA) that is supposed to be 40 times less bio accumulative than perfluorooctanoic acid (PFOA) [31]. Polysiloxane hydrophobic chemical was used as a softening agent with C_6-based fluorocarbon chemical in the coating solution to get advantageous functions of both water-repellent property as well as softening property. At first, different mixing ratios of C_6-based fluorocarbon chemical and polysiloxane hydrophobic softening agent were applied on one of the four PTFE laminated fabrics and then the best ratio on which the best results were obtained for this sample was selected for coating the rest three other PTFE laminated sample fabrics. After coating all four PTFE laminated three-layered fabrics, different test results were statistically analyzed in order to determine the significant or non-significant increases in the results of hydrostatic resistance, breaking strength, bending rigidity as well as water vapour permeability and air permeability. Water-repellent property of these fabrics was also evaluated after coating for its importance in outdoor sports clothing.

CHAPTER 2: LITERATURE REVIEW

The purpose of this chapter is to provide a broad overview of studies and theories that have been made in the field of research.

2.1 Waterproof breathable fabric

Waterproof breathable fabric incorporates two distinct functions in the fabric. One is waterproofness and another is breathability. This type of fabric provides the protection from rain, wind and at the same time maintains a comfortable microclimate just below the fabric layer. Breathability allows the water vapour transportation from one side of the fabric to another, while waterproofness restricts the transfer of water from outside to inside, protecting the users from getting wet. Hence, these two properties are two contrasting abilities. And development of waterproof breathable fabric is a big challenge as this fabric allows the transfer of water vapour and perspiration from inside of the fabric to outside and simultaneously restricts the passage of water from outside to inside [32].

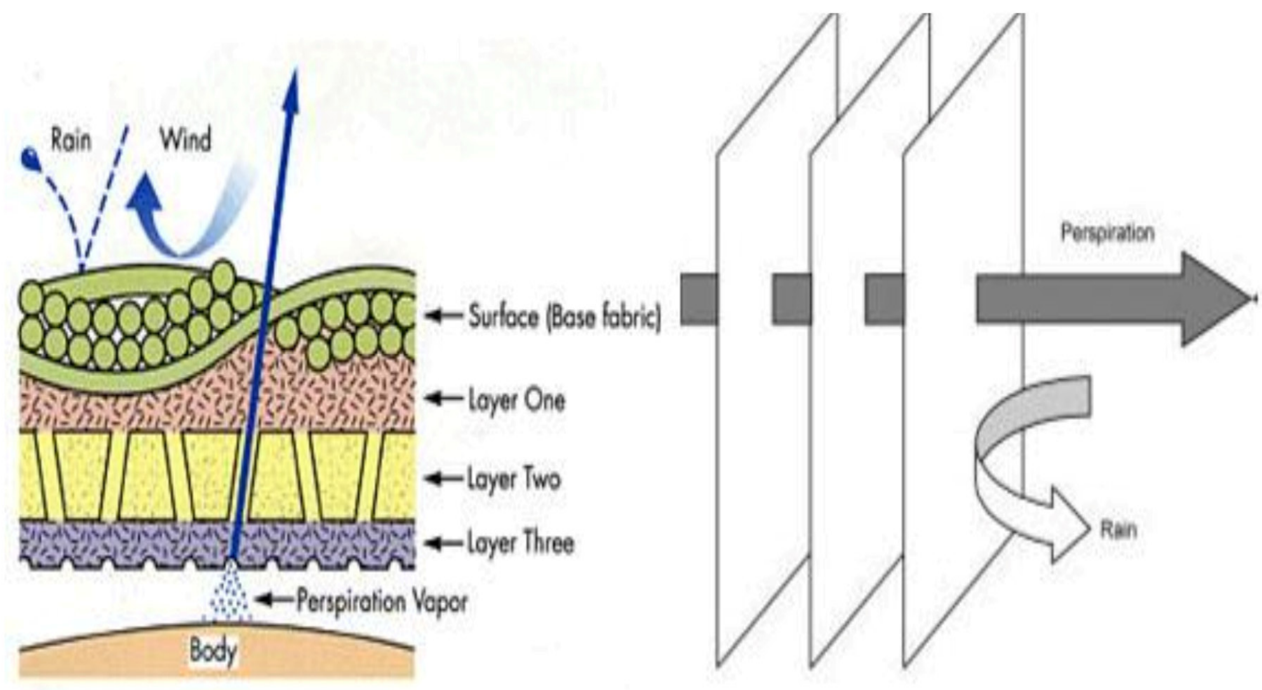

Figure 1. Transportation of water vapour through waterproof breathable fabric [33].

2.2 Waterproofness and breathability

Waterproofness and breathability are obviously the prime functions and most important features of waterproof breathable fabrics. A first level of classification of waterproof clothing contains the followings [34]:

- Conventional wet-weather clothing
- Clothing for sports and leisure
- Work clothing and uniforms including military uniform
- Personal protective clothing

2.2.1 Waterproofness

The term waterproofness means something that is impervious to water even under pressure. Waterproof fabric should entirely prevent the penetration and absorption of liquid water. Waterproofness is measured as the hydrostatic pressure needed to penetrate the waterproof fabric or as the hydrostatic resistance of the waterproof fabric until penetration. The standards used for determining waterproofness are as follows [34]:

- AATCC 127 : Water resistance – hydrostatic pressure test
- ISO 811 : Determination of resistance to water penetration – hydrostatic pressure test
- ASTM D 3393-91 : Standard specification for coated fabrics – waterproofness
- BS 3424-26 : Methods for determination of resistance to water penetration and surface wetting

Hydrostatic resistance value is measured in cmH_2O. And the values of 500 cmH_2O for high quality products and 130 cmH_2O for lower grade products have been reported [35].

2.2.2 Breathability

Breathability is defined by the ability of a fabric to allow water vapour to pass through it. If water vapour generated by the body cannot be released to the surrounding atmosphere, the relative humidity of the microclimate inside the clothing increases, as a result, the clothing becomes uncomfortable. The normal body core temperature is 37°C, while depending on conditions, skin temperature varies between 33°C and 35°C. The body is prevented from cooling at the same rate as heat is generated where perspiration cannot evaporate while liquid sweat is produced. This may happen during physical activity and hyperthermia might occur as the body core temperature increases. On the other hand, if heat loss exceeds the ability of the body to generate heat, body temperature decreases below normal levels. A decrease in core body temperature to 35°C or below is likely to stimulate hypothermia. This usually results from prolonged exposure to cold environment [36]. Hence it is very important for outdoor sports clothing to allow both the transmission of moisture vapour by diffusion so as to facilitate evaporative cooling and to provide sufficient insulation for hindering heat loss.

The heat energy produced during various activities and the perspiration required to provide adequate body temperature control have been studied. Table-1 shows the perspiration rates for activities ranging from sleeping to maximum work rate [7, 36, 37]

Table 1. Produced heat energy for various activities and corresponding perspiration rates [36]

Activity	Work rate (W/m^2 h)	Perspiration rate (mL/m^2 h)	Daily perspiration rate (g/m^2 per day)
Sleeping	40-60	20-99.08	450-2280
Gentle walking	185-200	330.28	7550-7600
Active walking with no pack	285-300	499.77	11480-11500
Active walking with light pack	385-400	660.56	15180-15200
Active walking with heavy pack	485-500	825.70	18900-19000
Mountain walking with heavy pack	650-810	986.50-1321.13	22700-30400
Maximum work rate	1100-1200	1664.45-1981.70	38300-45600

However, Breathability is evaluated based on water vapour transmission or evaporative resistance. Several methods are used to test the breathability of the fabrics like the followings [34]:

- The sweating hot plate method (evaporative resistance) - ISO 11092, ASTM F 1868
- The upright cup method - ISO 2528, ASTM E96
- The inverted cup method - ASTM E96
- The dynamic moisture permeation cell method - ASTM F 2298

Evaporative resistance (R_{et}) measures how breathable a fabric is in the unit of m^2Pa/W. The method developed by Hohenstein Institute using the Skin Model (ISO 11092) differs from the other methods. The values of R_{et} are widely used to compare various products. The values of R_{et} and expected performance are given in Table-2.

Table 2. Evaporative resistance (R_{et}) values with expected performance [36]

Range of R_{et} (m^2 Pa/ W)	**Expected performance**
0-6	Very good or extremely breathable; over 16000 g / m^2 per 24 hours. Comfortable at higher activity
6-13	Good or very breathable; 6000 to 15000 g / m^2 per 24 hours. Comfortable at moderate activity
13-20	Satisfactory or breathable; 4000 - 5000 g / m^2 per 24 hours. Uncomfortable at high activity
> 20	Unsatisfactory or slightly breathable; under 4000 g / m^2 per 24 hours. Moderate comfortable at low activity

2.3 Different methods of producing waterproof breathable fabrics

Waterproof breathable fabrics can be categorized into various types based on their manufacturing methods. Different methods involved in producing of waterproof breathable fabrics can be discussed into the following categories [38].

- Closely woven fabrics

- Microporous membranes or coatings
- Solid membranes or coatings
- Combination of microporous and solid membranes or coatings
- Smart breathable fabrics
- Incorporation of retro-reflective microbeads
- Fabrics based on biomimetics

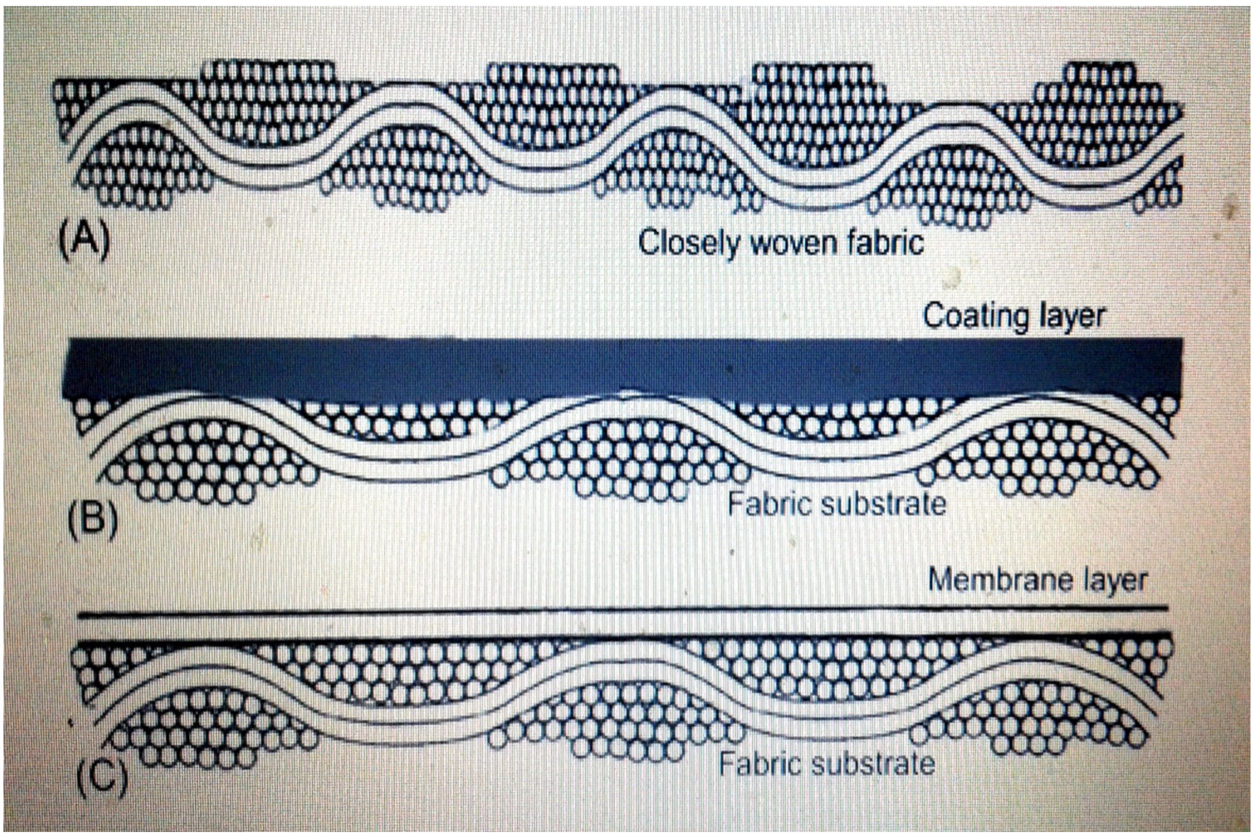

Figure 2. Major types of waterproof breathable fabrics: (A) closely woven fabric, (B) coated woven fabric and (C) laminated woven fabric [36].

2.3.1 Closely woven fabrics

Closely woven fabrics are produced from hydrophilic and absorptive yarns or from microfiber synthetic yarns that results in small pore size to give maximum protection against rain and wind. The concentration and surface area of inter yarn spaces should be as high as possible to maximize water vapour transportation through woven fabrics and the fabrics should be preferably prepared from hydrophilic and absorptive yarns. The ability of the fibers to undergo diffusion depends on the hydrophilic nature of the fibers and further liquid transmission is assisted by capillary transfer within the fiber bundle. Initially, these fabrics are not waterproof, but when come into contact with water, cotton fibers swell to such an extent that the inter yarn pores of the fabrics are significantly reduced and hence restrict the water passage. Inter yarn spaces and hydrophilic natures of fibers allow adequate water vapour permeability, but still air permeability is low. Ventile which is a famous waterproof breathable fabric is produced in this way. Long staple, combed and plied cotton yarns are woven to prepare this fabric using preferably oxford weave [39]. This ensures minimum pores in the fabric. When fabric surface is wetted by water, the cotton fibers swell transversely reducing pore size. High water pressure is required to penetrate the fabric. Densely woven fabric can also be produced from micro-denier synthetic filament yarns that have inherent water repellent properties. But, they do not swell when water is inserted, as a result, further coatings are required to obtain desirable results.

2.3.2 Microporous membranes or coatings

In microporous membranes or coatings, water vapour passes through open micro pores. Polyurethane, polytetrafluorothylene and acrylic are the most widely used elements. In this case, pore size becomes from 0.1 to 50 µm. If the maximum pore size at the outer surface of the barrier layer is about 2-3 µm or less, the waterproof properties of the fabric are usually sufficient. This microporous structure is air permeable and is capable of transmitting water vapour at physiologically accepted rates. A moisture vapour permeable waterproof coated fabric comprises of a base fabric and a synthetic microporous layer. The microporous layer is formed here from a coating solution of an organic solvent. In case of microporous membrane, holes are much smaller (2-3 µm) than the smallest rain drop (100 µm), yet very much larger than a water vapour molecule (40×10^{-6} µm) [17]. The microporous membranes and coatings operate on a similar principle. The water droplets cannot penetrate the micro pores of the membranes and coatings while the moisture vapour molecules are pushed through.

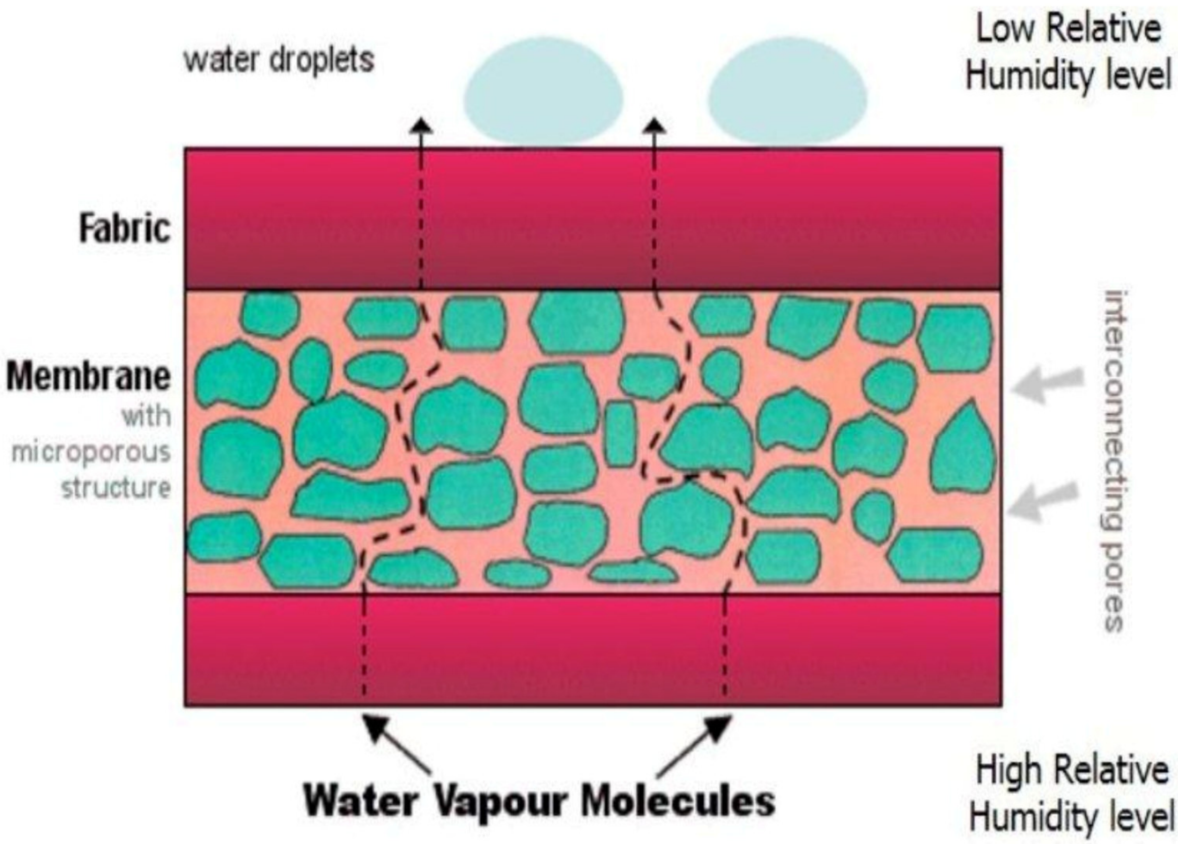

Figure 3. Transmission of water vapour molecules through microporous structure [40].

Methods of developing microporous membranes and coatings are as below [38]:

- Mechanical fibrillation
- Wet coagulation process
- Thermo coagulation process
- Foam coating
- Solvent extraction
- Melt blown technology
- Point bonding technology
- Radio frequency (RF) / ion / UV or E beam radiation

2.3.3 Solid membranes or coatings

Solid membranes or coatings consist of modified polymers. They are usually hydrophilic films without any pores or holes. Here, water vapour transmission is occurred by molecular diffusion or by chemical adsorption process. Amorphous region is formed in the main polymer system of the hydrophilic part and this amorphous region acts as intermolecular pores allowing water vapour molecules to pass through, but, still prevents the penetration of liquid water due to solid nature [38, 41].

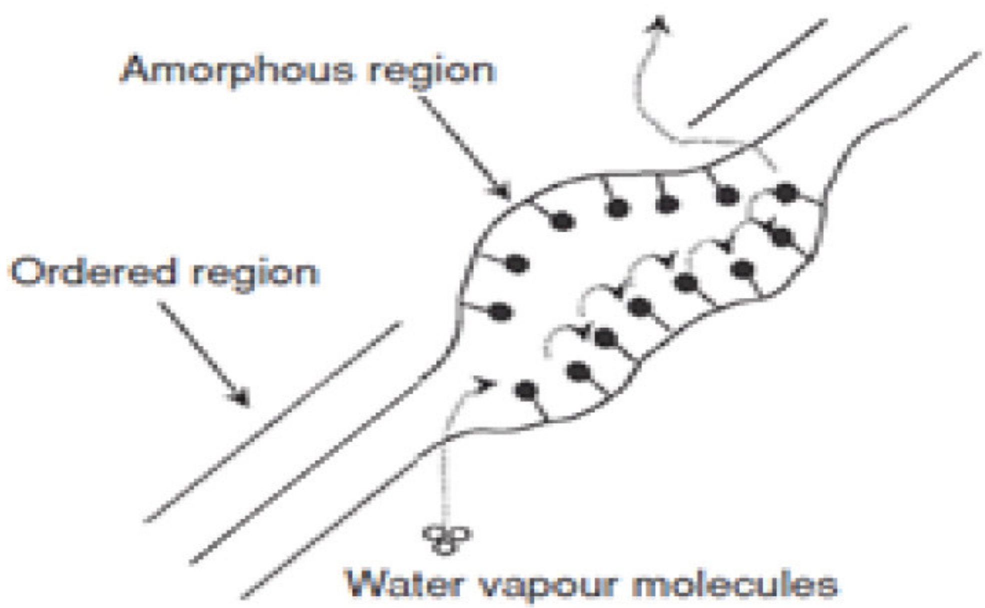

Figure 4. Transmission of water vapour molecules through hydrophilic structure [33].

2.3.4 Combination of microporous and solid membranes or coatings

Combination of microporous and hydrophilic membranes or coatings is another method of preparing waterproof breathable fabric. The microporous mesh or material is imbued with a hydrophilic material in case of membranes. Hydrophilic finishes are applied over microporous films that have been attached to the fabric in case of coatings. And, this ensures enhanced waterproofing capacity without hampering the breathability to a large extent [42].

2.3.5 Smart breathable fabrics

By stopping the transfer of vapour and heat at low temperature and at high temperature, shape memory polymer restricts the loss of body warmth in case of smart breathable fabrics. Here, more water vapour and heat are transferred from inside to outside of the fabric than ordinary waterproof breathable fabrics. The coating substance on the fabric exists in a swollen state at lower temperature by absorbing water from the surroundings. This results in the closure of micro cracks. At a temperature higher than the transition temperature, the coating exists in a collapsed state due to the predominance of hydrophobic interactions resulting in opening of the micro cracks. The phase change material of the fabric delays the transient response when a sudden change in ambient condition occurs and decreases body heat loss [33].

However, some other techniques, i.e., incorporation of retro-reflective microbeads and fabrics based on biomimetic have been developed for obtaining improved properties of breathable waterproof fabrics.

2.4 Laminated waterproof breathable fabrics

Laminated waterproof breathable fabric consists of one or more textile substrates that are combined with membrane by using adhesives or by using heat and pressure. Membranes are thin materials made from polymeric substances that offer high resistance to water penetration but allow water vapour at the same time. These membranes are of two types [33]:

- Microporous membranes
- Hydrophilic membranes

Microporous membranes have tiny holes on their surface smaller than a rain drop, but larger than a water vapour molecule. Mostly these types of membranes are of hydrophobic character. Some of these membranes are made from polytetrafluoroethylene polymer, polyurethane, polyvinylidene fluoride etc. Again, hydrophilic membranes are tight membranes, mainly of polyester but also of polyurethane [43].

2.4.1 Microporous polyurethane membrane

Polyurethane (PU) membranes are popular hydrophobic microporous polymeric membranes. These membranes are made by the process of a controlled phase separation from three or multi-component systems (pre-polymer, solvent or a mixture of these and non-solvent). The phase separation is a process in which the homogeneous pre-polymer solution in specified solvents changes into a gel to form a macromolecular network of polymer with a liquid phase dispersed in its structure. Such a solution is unstable and can be changed into a two-phase system under the influence of various factors; one of the phases so produced is characterized by a high polymer concentration and the other by a low polymer concentration. The phase that is richer in polymer is solidified after the phase separation to form a microporous membrane. The properties of such a

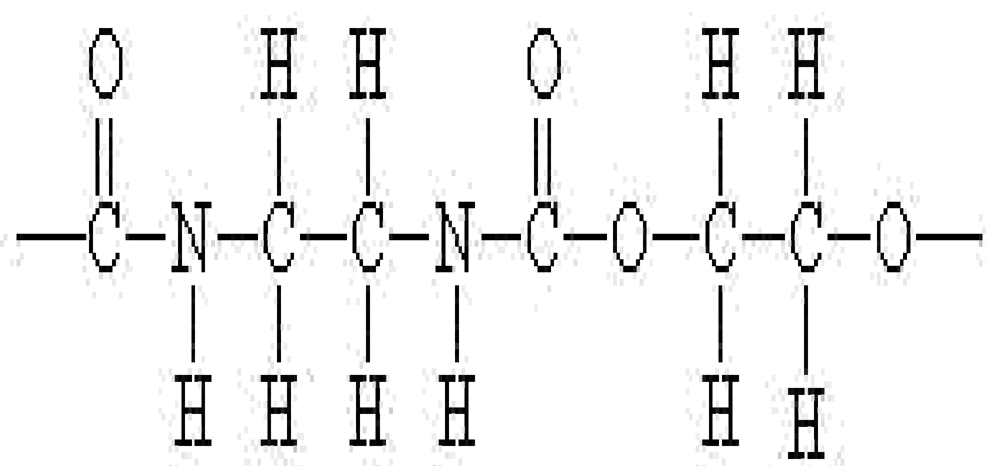

Figure 5. Structure of polyurethane [44].

membrane depend on the condition of phase separation and solidification. The phase separation of polymeric solutions can be induced by many factors, with the change in the composition of membrane forming solution being the most frequently used factor to cause inversion. Considering the factor that causes the phase separation, several methods for producing of microporous polymeric membranes can be distinguished. The most important of which in the case of the membranes used in textile systems is the separation induced by solvent extraction and solvent evaporation. These methods are also known as wet and thermal coagulations. In both coagulation methods, the micro-capillaries are produced by selective removal of the solvent of pre-polymers and non-solvent [13].

2.4.2 Microporous polytetrafluoroethylene membrane

Microporous polytetrafluoroethylene (PTFE) membrane is widely used in the textile industry. Hydrophobic microporous PTFE membrane is produced by the drawing process under critical conditions from tight or leak-proof membranes which results in the formation of numerous micro-cracks or micro-porosity. PTFE membranes are known under the general trade name of Gore-Tex from the American firm W.L. Gore & Assoc. Inc. [43].

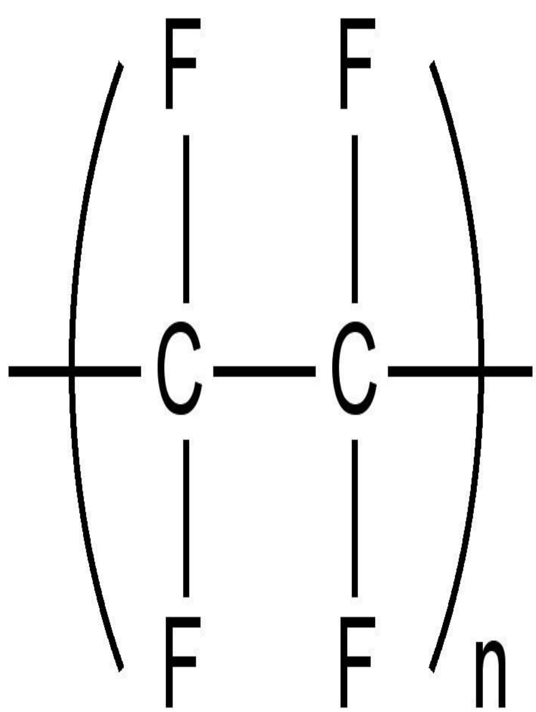

Figure 6. Structure of polytetrafluoroethylene [45].

2.5 Water-repellent fabrics and coating

Water-repellent property of a textile fabric is the ability of the material to resist being wetted. In this case, drops of water should not spread on the surface of the fabric and should not make the fabric wet. The drops should stay on the surface and easily roll off the fabrics. A water-repellent fabric can be achieved, when the fibers are usually coated with a hydrophobic type of compound and the pores are not filled in the course of the treatment. Water-repellent surface allows water vapour. Air permeability of the water-repellent fabric is not significantly reduced [31]. This situation provides the clothing comfort. Water-repellent fabrics have many uses including industrial, consumer and apparel purposes. Water-repellent chemical makes the fabric water-repellent by the formation of polymeric chains and graft bonds inside the textile structure [46].

2.5.1 Fluorocarbon and polysiloxane chemicals

Fluorocarbon is the general name for most of organic fluorine compounds. They are synthetic compounds that contain carbon and fluorine. There has been a market increase in the commercial use of fluorochemicals in recent years, particularly to impart water-repellent property to the fabric. Various types of fluorochemicals are used as textile finishing chemicals in order to impart water- repellent property [46].

Fluorocarbon provides advantages to textile materials like the followings [31]:

- textile materials are protected against water and stain
- it is resistant to washing
- original colors of the fabrics are protected
- allows water vapour permeability
- air permeability is not significantly reduced

Because of this wide and growing use of fluorochemicals, there are some different and sometimes conflicting views as to the most efficient and effective product among all fluorochemical based water-repellent chemicals. It consists of perfluorinated carbon chains incorporating a polymer backbone with perfluoro groups as its side chains [47].

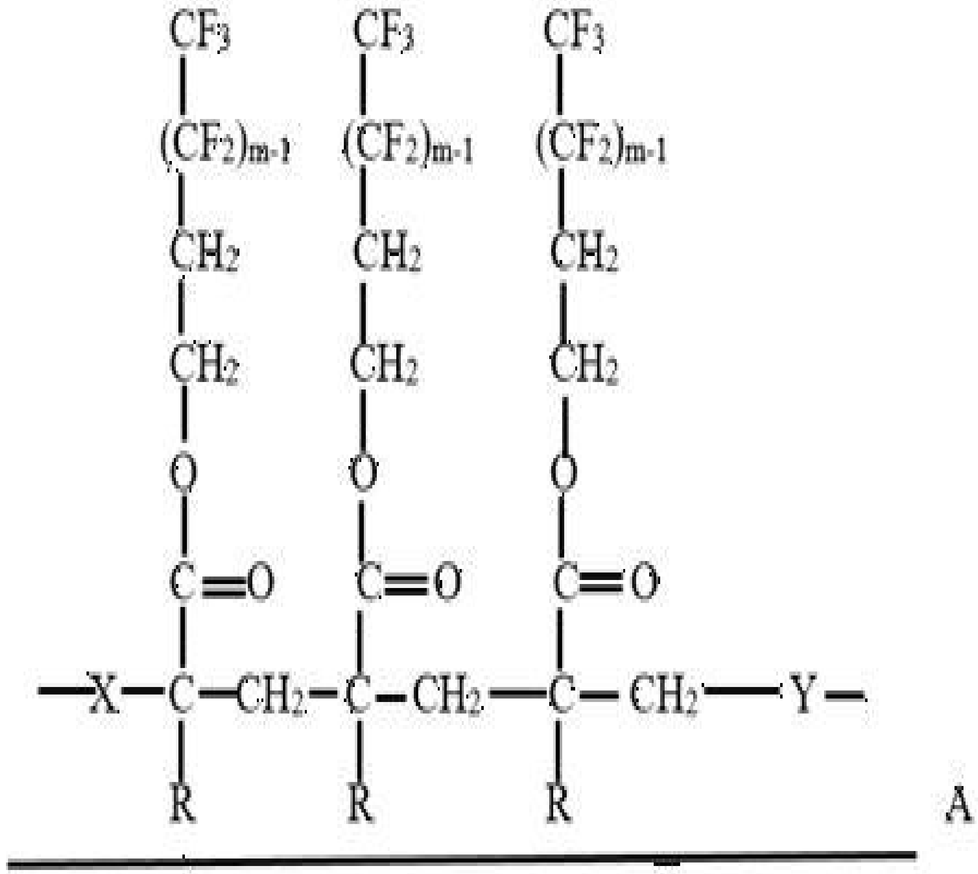

Figure 7. Fluorocarbon repellency on fiber surface. X & Y are co-monomers, R=H or CH_3 and A is the fiber surface.

Some existing fluorochemicals are made with C_8 carbon backbone chains that can release perfluorooctanesulfonate (PFOS), perfluorooctanoic acid (PFOA) and other toxic and hazardous materials. Hence, C_6-based fluorocarbon chemical has been introduced and this chemical is environment friendly producing perfluorohexanoic acid (PFHA) that is supposed to be 40 times less bio accumulative than perfluorooctanoic acid (PFOA) [31].

Again, silicone based polysiloxane hydrophobic softening agent can introduce two distinct properties, i.e., water-repellent property and softening property. The mechanism of polysiloxane resin coating is shown in Figure-8.

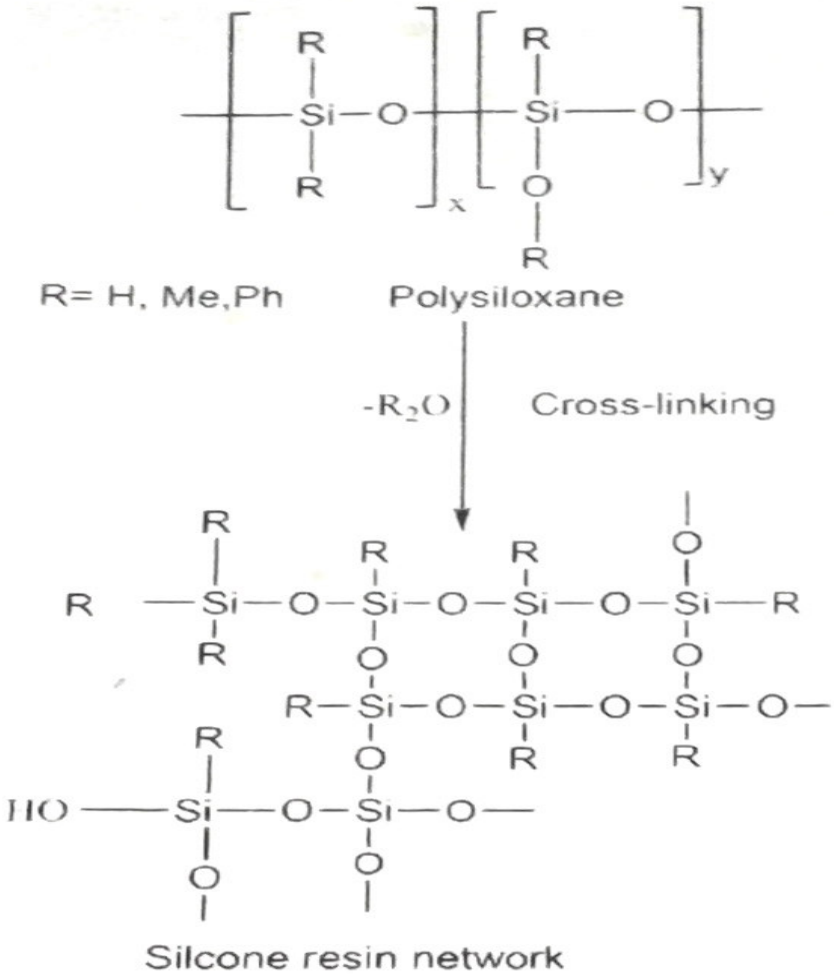

Figure 8. Mechanism of coating with polysiloxane [48].

2.5.2 Methods of Coating

Different types of coating methods are as below:

- Pad-dry-cure coating
- Direct coating
- Calendar coating
- Hot melt extrusion coating
- Foam finishing coating

2.5.2.1 Pad-dry-cure coating

This fabric coating technique is regarded as a textile finishing technique and this technique can be used to add a variety of coatings. This coating is also simply referred to as padding. Pad-dry-cure coating most often refers to a fiber coating used for the application of both micro and nano materials or even chemical compositions. In this process, the fabric is submerged in the coating solution. Excess liquid solution is then squeezed out in the rollers. And finally the fabric is dried and cured [49].

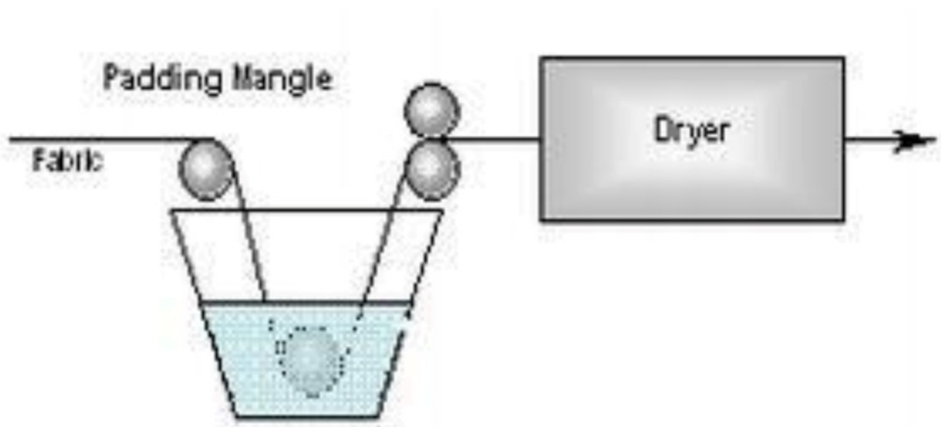

Figure 9. Pad-dry-cure coating.

2.5.2.2 Direct coating

Direct coating is usually carried out on tightly woven fabrics with smooth surfaces. This coating process is done by using the knife mechanism. The thickness of coating depends on the gap between the knife and the surface. [39].

2.5.2.3 Calendar coating

In this method, the coating films are created from polymer dough. The calendars evenly spread the dough over the fabric substrate using pressure. The calendars can be made up from a number of rollers [50].

2.5.2.4 Hot melt extrusion coating

For coating thermoplastic polymers, hot melt extrusion coating can be used. Polymer granules are fed between heated rollers. When heated, the granules melt and spread onto the substrate [51].

2.5.2.5 Foam finishing coating

Foam coating is used on substrates that cannot be directly coated due to non-smooth surfaces. This method also helps to maintain the handle and drape properties of the fabric [50]. Foam finishing coating is useful during coating of heavy fabrics, such as carpets, and this process can be used effectively to coat only one side of a fabric material [49].

CHAPTER 3: EXPERIMENTAL

This chapter has been discussed into two parts. In the first part, methodology for investigation of hydrostatic resistance and comfort properties of PU membrane laminated fabrics has been discussed. In the second part, methodology for enhancement of hydrostatic resistance and mechanical performance of PTFE membrane laminated fabrics has been discussed.

3.1 Methodology for investigation of hydrostatic resistance and comfort properties of PU membrane laminated fabrics

3.1.1 Materials

In order to produce two-layered and three-layered laminated fabrics, three different types of polyester plain woven fabrics with different fabric weights and different warp and weft cover factors were used as outer layers. One type of single jersey polyester knitted fabric with 127 GSM (g/m^2) was applied as an inner layer for preparation of each three-layered laminated fabric. 20 GSM (g/m^2) microporous polyurethane (PU) membrane was used as an inner layer for each two-layered fabric lamination and as a middle layer for each three-layered fabric lamination. Polyester woven and polyester knitted fabrics were purchased from Hira Mukta Design & Fashion of Bangladesh. PU membrane was obtained from Nanotex Group in Czech Republic. Particulars of three different types of woven fabrics and one type of knitted fabric are given in Table-3.

Table 3. Particulars of woven and knitted fabrics

Fabric code	Type of fabric	Fabric weight (g/m^2)	Warp and weft cover factor of woven fabric (K_1 & K_2)	Stitch density of knitted fabric (stitches/cm^2)
F-1	Polyester plain woven fabric	139	(14 & 12)	------
F-2	Polyester plain woven fabric	128	(13 & 10)	------
F-3	Polyester plain woven fabric	122	(11 & 10)	------
K	Polyester single jersey knitted fabric	127	------	208

3.1.2 Methods

3.1.2.1 Lamination process

Comel PL/T 1250 Heat and Press Machine was used for producing six different types of laminated fabrics. Two-layered three samples were prepared placing one upon another in the order of PU membrane and each of three different polyester woven fabrics separately. And three-layered three samples were prepared laying one upon another in the order of each of three different polyester woven fabrics separately, PU membrane and polyester knitted fabric. Then fabrics were laminated under heat and pressure treatment of the machine at 160°C temperature with 2 bar pressure for 15 seconds. Characteristics of produced six laminated sample fabrics are shown in Table-4.

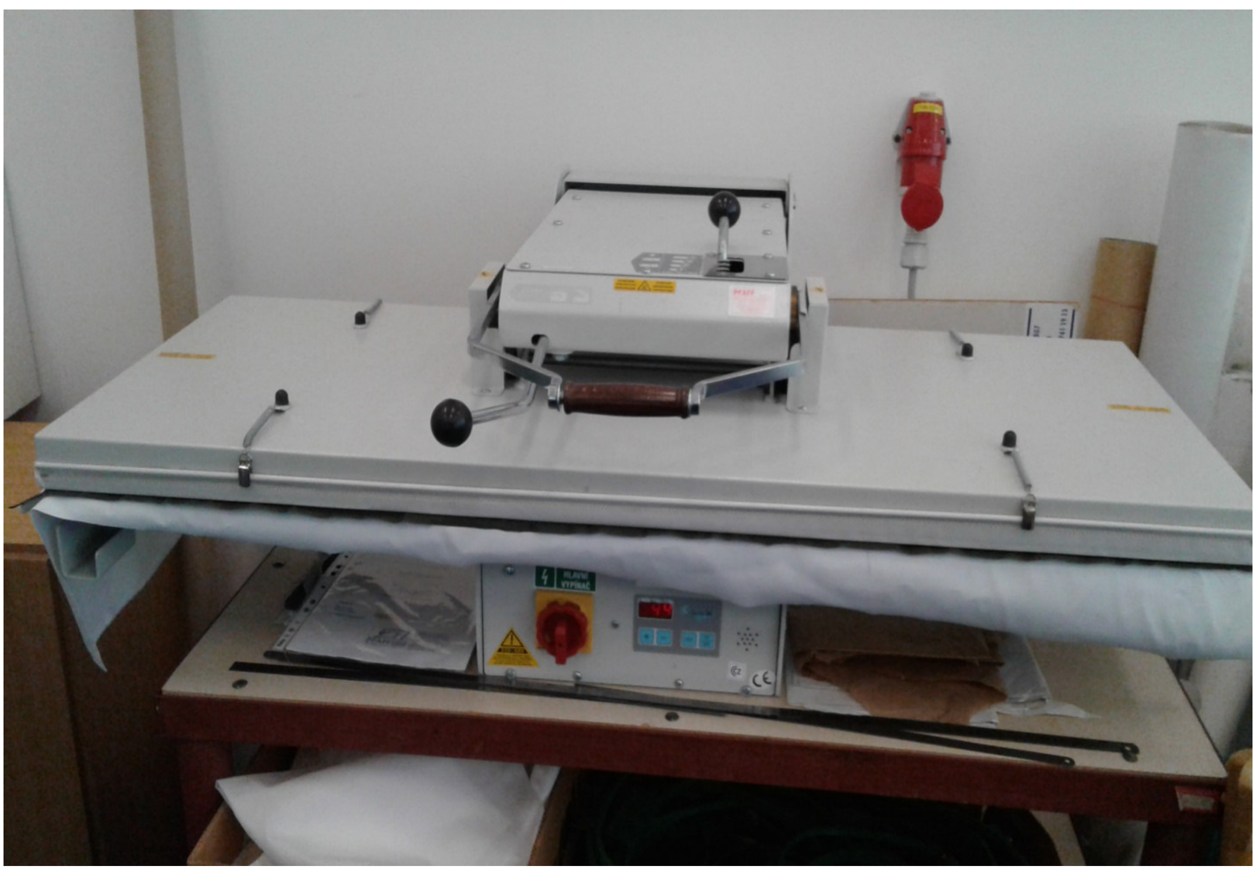

Figure 10. Lamination by Comel PL/T 1250 Heat and Press Machine.

Table 4. Characteristics of PU membrane laminated sample fabrics

Sample Fabric code	**Layered structure of fabric (outer layer to inner layer)**	**Areal density of sample fabric (g/m^2)**	**Thickness of sample fabric (mm)**	**Density of sample fabric (kg/m^3)**
		(Mean ± SD)	(Mean ± SD)	(Mean ± SD)
FM-1	F-1 + PU membrane	158 ± 1.01	0.42 ± 0.01	376.19 ± 1.16
FM-2	F-2 + PU membrane	147 ± 1.29	0.40 ± 0.01	367.50 ± 1.26
FM-3	F-3 + PU membrane	141 ± 1.12	0.39 ± 0.01	361.53 ± 1.18
FMK-4	F-1 + PU membrane + K	283 ± 1.77	0.69 ± 0.01	410.14 ± 1.55
FMK-5	F-2 + PU membrane + K	271 ± 1.42	0.68 ± 0.01	398.53 ± 1.61
FMK-6	F-3 + PU membrane + K	263 ± 1.26	0.67 ± 0.01	392.54 ± 1.85

3.1.2.2. Characterization of laminated fabrics

Cover factor

Warp cover factor and weft cover factor of outer woven layer part of the laminated fabric were measured using the Peirce equation [52]:

$$K_1 = n_1 / (N_1)^{1/2} \text{ and } K_2 = n_2 / (N_2)^{1/2} \quad (1)$$

Here, 'K_1' is warp cover factor, 'K_2' is weft cover factor, 'n_1' is warp yarn density/inch, 'n_2' is weft yarn density/inch, 'N_1' is English count of warp yarn and 'N_2' is English count of weft yarn.

Stitch density

Stitch density of inner knitted layer part of the laminated fabric was calculated by the multiplication of courses/cm and wales/cm using optical microscope [53].

Fabric weight

Fabric weight per unit area was measured using electronic weighing scale according to CSN EN 12127 [54].

Fabric thickness

Fabric thickness was measured according to EN ISO 5084 [55] at a pressure of 100 Pa with Louis Schopper Automatic Micrometer.

Fabric density

Fabric density of the laminated sample fabric was calculated by the following equation [56]:

$$\text{Fabric density} = \frac{W}{t}\ [\mathrm{Kg/m^3}] \tag{2}$$

Here, 'W' is fabric mass per unit area and 't' is fabric thickness.

3.1.2.3 Morphology

The morphological cross-sections of the laminated samples show the layered structures of the fabrics. This morphology was examined using high resolution of scanning electron microscope VEGA TS 5130- TESCAN that was used in the previous study [57]. To increase the surface conductivity, the samples were sputter coated with gold.

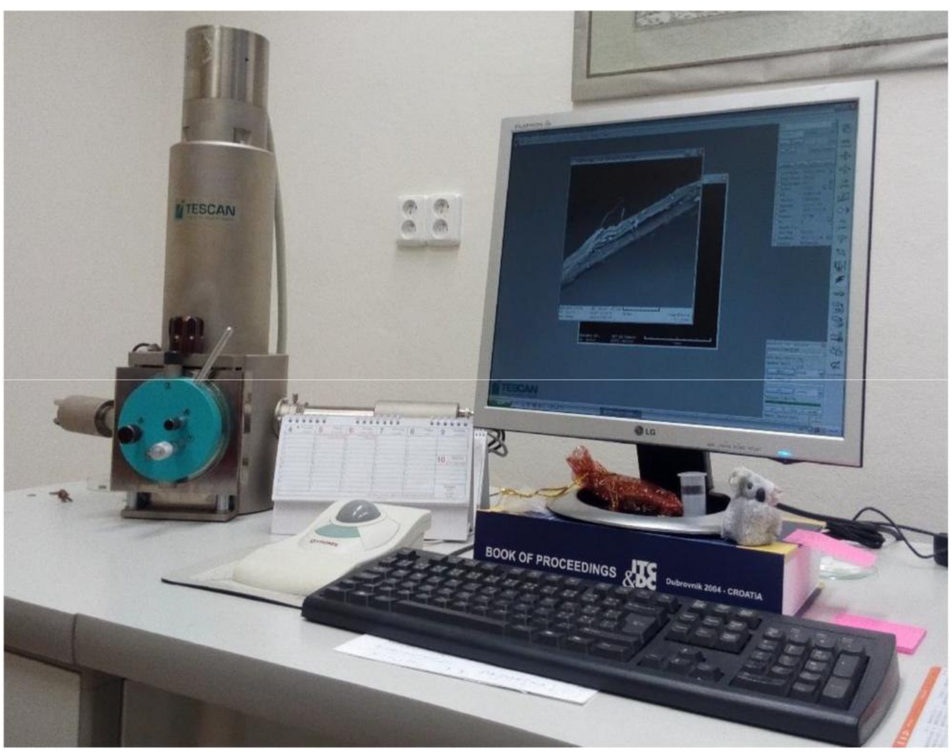

Figure 11. VEGA TS 5130-TESCAN Scanning Electron Microscope.

3.1.2.4 Hydrostatic resistance

Hydrostatic resistance tests were carried out by SDL ATLAS Hydrostatic Head Tester Model MO18 according to AATCC 127 [58] at 20±2ºC. The rate of increase of water pressure per minute was kept at 60±3 cmH_2O. Water pressure was recorded at the point when water penetrated from outer layer to inner layer showing three drops of water or crack of sample or constant fall of water pressure. The unit was expressed as cmH_2O and obtained test results are reported in Table-5.

Figure 12. MO18 Hydrostatic Head Tester.

Table 5. Properties of PU membrane laminated sample fabrics

Properties		FM-1	FM-2	FM-3	FMK-4	FMK-5	FMK-6
Hydrostatic resistance (cmH_2O)	Mean	675	639	615	829	786	758
	SD	2.49	2.16	2.45	2.94	2.62	3.09
	UL	678	641	618	832	789	761
	LL	673	637	612	826	783	754
Evaporative resistance, R_{et} (m^2Pa / W)	Mean	7.44	6.82	6.40	9.18	8.68	8.22
	SD	0.16	0.12	0.11	0.18	0.10	0.12
	UL	7.58	6.92	6.50	9.34	8.77	8.32
	LL	7.30	6.72	6.30	9.02	8.59	8.12
Relative water vapour permeability, RWVP (%)	Mean	45.42	47.34	48.62	39.42	41.02	42.92
	SD	0.27	0.28	0.23	0.25	0.29	0.22
	UL	45.66	47.59	48.82	39.64	41.28	43.12
	LL	45.18	47.09	48.42	39.20	40.76	42.72
Air permeability ($l/m^2/s$)	Mean	1.97	2.38	2.59	1.42	1.73	1.85
	SD	0.03	0.04	0.06	0.03	0.04	0.05
	UL	1.99	2.40	2.63	1.44	1.75	1.88
	LL	1.96	2.35	2.55	1.40	1.71	1.82
Thermal resistance (10^{-3} Km^2/W)	Mean	13.84	12.20	10.94	19.36	17.54	16.56
	SD	0.17	0.21	0.19	0.22	0.27	0.16
	UL	13.99	12.38	11.10	19.56	17.78	16.70
	LL	13.69	12.02	10.78	19.16	17.30	16.42
Bending rigidity (10^{-6} Nm)	Mean	12.67	12.08	11.79	21.31	20.91	20.57
	SD	0.07	0.10	0.06	0.07	0.04	0.04
	UL	12.75	12.19	11.86	21.39	20.95	20.62
	LL	12.58	11.96	11.71	21.23	20.86	20.52
Breaking force (N)	Mean	373	354	342	451	423	405
	SD	4.32	4.67	3.92	4.94	5.31	4.63
	UL	376	358	345	455	428	409
	LL	369	350	338	447	419	401

SD: Standard deviation; UL: Upper limit; LL: Lower limit

3.1.2.5 Water vapour permeability

The water vapour permeability of the sample fabric was measured by PERMETEST instrument. This instrument is able to determine non-destructive measurement of the samples according to ISO 11092 standard [59] and it works on the principle of heat flux sensing. The fabric sample was placed on a measuring head over a semi-permeable foil and exposed to parallel air flow at a velocity of 1 m/s. The measurement was carried out at room temperature for isothermal conditions following the skin model [60]. When water flows into the measuring head, some amount of heat is lost and the instrument measures the heat loss from the measuring head due to evaporation of water without fabric and with fabric. The relative water vapour permeability (RWVP) of the sample is calculated by the ratio of heat loss from the measuring head with fabric (q_s) and heat loss from the measuring head without fabric (q_o) as below equation [61, 62].

$$RWVP = (q_s / q_o) \times 100\% \quad (3)$$

PERMETEST enables to determine water vapour transmission of the fabric by measuring the two parameters as RWVP% and evaporative resistance (R_{et}) in the unit of m^2Pa/W. Five tests for each sample were done and the mean test results are presented in Table-5.

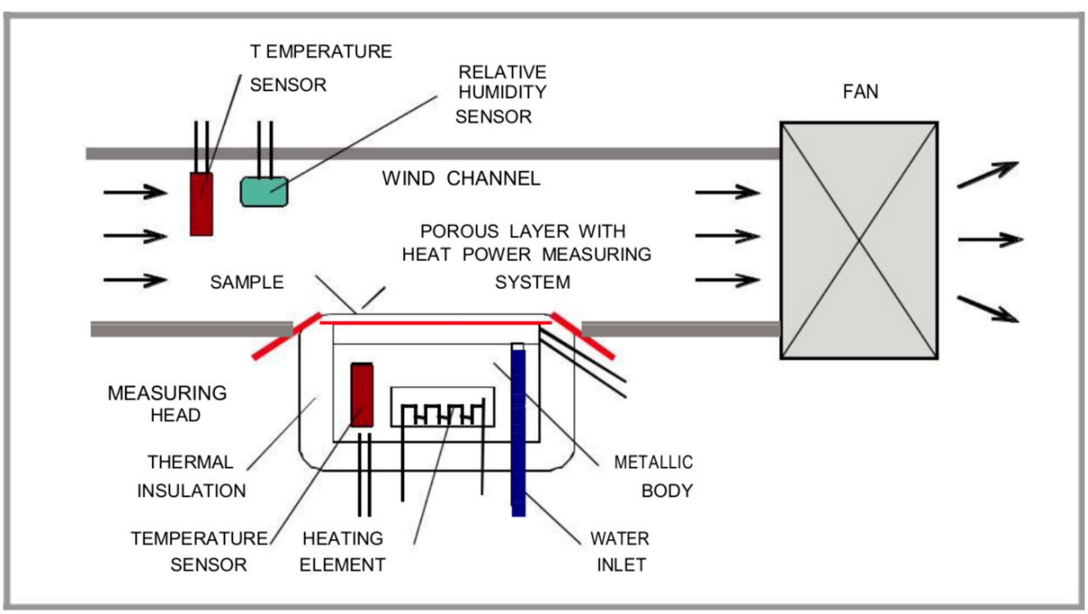

Figure 13. PERMETEST skin model.

3.1.2.6 Air permeability

The air flow rate that passes perpendicularly under a prescribed air pressure through a known area between the two surfaces of a material is considered as air permeability. Textest FX-3300 air permeability tester was used to measure air permeability of the samples according to standard EN ISO 9237 [63]. Test area of the sample was 20 cm^2 and air pressure difference between the two surfaces was kept at 200 Pa because of the layered structures of the sample fabrics. Average value of ten measurements was taken for each sample in the unit of $l/m^2/s$ and the results are shown in Table-5.

3.1.2.7 Thermal resistance

Thermal property, like, thermal resistance was measured by ALAMBETA apparatus which is a computer controlled instrument for measuring the basic static and dynamic thermal characteristics of textiles. The contact pressure was 200 Pa in all cases, and the CV (coefficient of variation) values of all the samples were lower than 4% as like as used in other experiment [64]. The values of thermal resistance of the laminated fabrics were determined from the instrument and test results are given in Table-5. Measurement of thermal property by ALAMBETA is standardized in the Internal Standard of Textile Faculty of Technical University of Liberec [65].

3.1.2.8 Bending rigidity

By TH-7 instrument, bending force of the sample fabric was directly obtained. Bending rigidity was calculated by the multiplication of this bending force value with a constant value of (0.7×10^{-6}). Here, unit was expressed as Nm. The device TH-7 was developed in the Department of Textile Evaluation at Technical University of Liberec. It was developed by means of innovation of device TH-5 on which only rectangle samples sized 2.5x5 cm could be measured. The following features give the differences between the model of device TH-5 and TH-7 [66]:

- The clamping and sensor jaw has been extended so that the device can be used for measuring rectangular, square and circular samples.

- The revolving clamping jaw has been designed so that it can turn in both directions, which enables one to draw the whole hysteresis loop of bending.
- The sensor jaw has been adjusted so that the bending power can be scanned in both directions: face– face and back–back. The sensor jaw is of shape U.

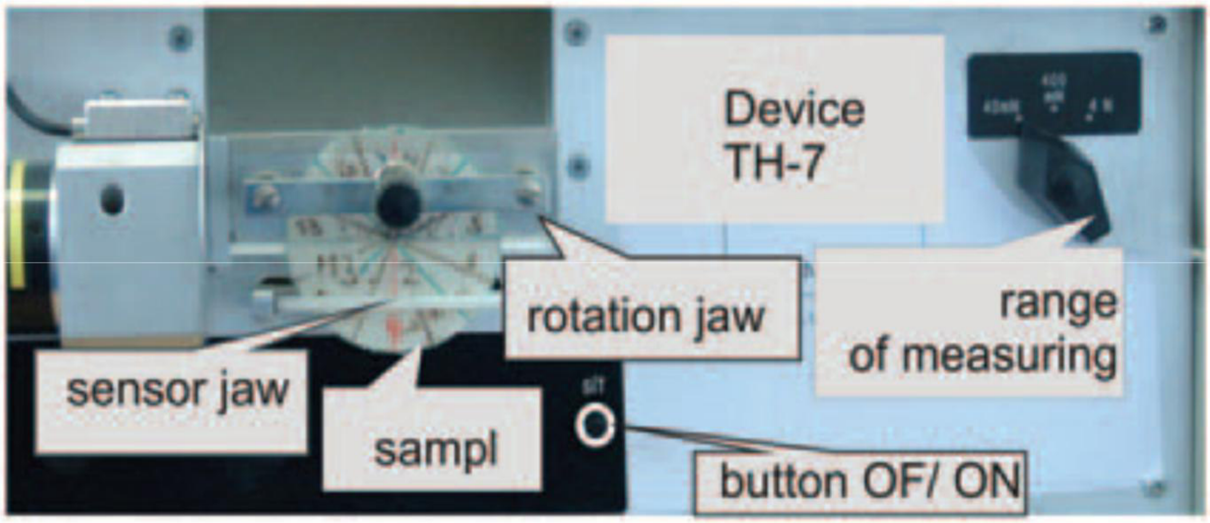

Figure 14. Device of TH-7.

- Teflon tubes on the sensor jaw reduce the coefficient of friction between the tube and the bent fabric.
- New software has been developed in order to control the device and store the measured data.
- To set 10 cycles of automatic bending at maximum is possible. The values of every cycle as well as the final average value are recorded.

3.1.2.9 Breaking strength

Testometric M350-5CT machine (UK) was used at room temperature according to CSN EN ISO 13934-1 [67] for breaking strength test of the samples. Specimen size was kept at 20cm x 5cm and testing speed was 100 mm/ min. The unit was expressed as breaking force in Newton (N) and the test results are given in Table-5.

3.2 Methodology for enhancement of hydrostatic resistance and mechanical performance of PTFE membrane laminated fabrics

3.2.1 Materials

3.2.1.1 Waterproof breathable laminated fabrics

Four different types of polytetrafluoroethylene (PTFE) hydrophobic microporous membrane laminated waterproof breathable fabrics were used in this part of the experiment. The fabrics were purchased from Hira Mukta Design & Fashion of Bangladesh. All the samples were three-layered fabrics. Outer layers of all four samples were with polyester plain woven structures. Inner layers of first two samples were with polyester knitted structures and inner layers of third and fourth samples were with polyester fleece knitted structures. PTFE membrane was laminated as a middle layer for each sample. Particulars of the laminated sample fabrics are shown in Table-6.

Table 6. Particulars of PTFE membrane laminated sample fabrics

Fabric sample code	**Fabric construction (outer layer to inner layer)**	**Warp and weft cover factor of outer woven part of fabric (K_1 & K_2)**	**Stitch density of inner knitted part of fabric (stitches/cm^2)**
WMK-1	Polyester plain woven + PTFE membrane + polyester knitting	(10 & 7)	273
WMK-2	Polyester plain woven + PTFE membrane + polyester knitting	(19 & 14)	925
WMF-3	Polyester plain woven + PTFE membrane + polyester fleece knitting	(15 & 12)	192
WMF-4	Polyester plain woven + PTFE membrane + polyester fleece knitting	(15 & 13)	221

3.2.1.2 Coating chemicals

®RUCOSTAR EEE6 with density of 1.03 g/cm^3 at 20°C was used as a water-repellent chemical. This chemical is a C_6-based fluorocarbon chemical which is different from conventional C_8-based fluorocarbon chemical. It is ecologically optimized agent for water and free from perfluorooctanoic acid (PFOA), perfluorooctanesulfonate (PFOS) and alkylphenol ethoxylate (APEO). ®RUCOFIN HSF polysiloxane hydrophobic softening agent was added with ®RUCOSTAR EEE6 in the coating solution. This chemical is easily diluted with water and its density is 1.10 g/cm^3 at 20°C. Both of the chemicals were purchased from Rudolf GmbH, Germany.

3.2.2 Methods

3.2.2.1 Coating process

At first, WMK-2 sample was considered as a standard sample among these four samples and this sample was coated with different mixing ratios of C_6-based fluorocarbon resin (®RUCOSTAR EEE6) and polysiloxane hydrophobic softening agent (®RUCOFIN HSF) according to T-2, T-3, T-4, T-5, T-6 and T-7. Characteristics of WMK-2 sample fabric coated with different ratios of ®RUCOSTAR EEE6 and ®RUCOFIN HSF are shown in Table-7. And properties of WMK-2 sample fabric coated with different ratios of ®RUCOSTAR EEE6 and ®RUCOFIN HSF are shown in Table-8. Then after analysis, the best ratio, from which the best results were obtained, was selected and applied for coating the rest three sample fabrics, like, WMK-1, WMF-3 and WMF-4 samples. However, pad-dry-cure method was used for coating. The homogeneous coating solution was made by ordinary manual stirring and put into the coating bath. The waterproof fabrics were impregnated in the coating bath at room temperature. The coating fluid was agitated by glass rod to have uniform coating on the fabrics. After impregnation, the fabrics were passed through the squeezing rollers. Then the fabrics were dried and finally cured at 160°C for 1 minute.

Table 7. Characteristics of WMK-2 sample fabric coated with different ratios of ®RUCOSTAR EEE6 and ®RUCOFIN HSF

Fabric sample		Areal density of Fabric (g / m^2)	Fabric thickness (mm)	Fabric density (Kg / m^3)	Increase in areal density after coating (%)
T-1 (uncoated)	Mean	167	0.350	477.14	-----
	SD	2.29	0.01	2.20	
T-2 (40 g/L ®RUCOSTAR EEE6 with 15 g/L ®RUCOFIN HSF)	Mean	182	0.377	482.76	8.98
	SD	2.06	0.01	2.05	
T-3 (50 g/L ®RUCOSTAR EEE6 with 15 g/L ®RUCOFIN HSF)	Mean	185	0.380	486.84	10.78
	SD	2.19	0.01	3.15	
T-4 (60 g/L ®RUCOSTAR EEE6 with 15 g/L ®RUCOFIN HSF)	Mean	177	0.370	478.38	5.99
	SD	2.03	0.01	3.30	
T-5 (40 g/L ®RUCOSTAR EEE6 with 20 g/L ®RUCOFIN HSF)	Mean	183	0.378	484.13	9.58
	SD	2.35	0.01	3.47	
T-6 (50 g/L ®RUCOSTAR EEE6 with 20 g/L ®RUCOFIN HSF)	Mean	180	0.374	481.28	7.78
	SD	2.37	0.01	3.28	
T-7 (60 g/L ®RUCOSTAR EEE6 with 20 g/L ®RUCOFIN HSF)	Mean	176	0.368	478.26	5.39
	SD	2.39	0.01	3.33	

Table 8. Properties of WMK-2 sample fabric coated with different ratios of ®RUCOSTAR EEE6 and ®RUCOFIN HSF

Properties		T-1	T-2	T-3	T-4	T-5	T-6	T-7
Hydrostatic resistance (cmH_2O)	Mean	1522	1672	1712	1633	1686	1654	1624
	SD	6.13	6.80	7.36	6.55	7.41	6.34	6.48
Breaking force (N)	Mean	478	559	572	514	562	535	504
	SD	7.69	8.27	8.36	8.09	8.47	7.82	8.26
Bending rigidity (10^{-6} Nm)	Mean	13.58	14.51	14.63	14.16	14.48	14.19	14.04
	SD	0.12	0.11	0.13	0.22	0.14	0.21	0.16
Evaporative resistance, R_{et} (m^{2}Pa / W)	Mean	8.42	8.74	8.84	8.54	8.78	8.66	8.52
	SD	0.27	0.21	0.31	0.22	0.25	0.23	0.24
Relative water vapour permeability, RWVP (%)	Mean	42.74	42.46	42.40	42.56	42.44	42.50	42.58
	SD	0.23	0.29	0.36	0.31	0.38	0.32	0.33
Air permeability (l/m^2/s)	Mean	0.72	0.69	0.68	0.70	0.68	0.69	0.70
	SD	0.05	0.04	0.05	0.03	0.04	0.05	0.04

After coating WMK-1, WMK-2, WMF-3 and WMF-4 samples on the basis of best ratio of ®RUCOSTAR EEE6 and ®RUCOFIN HSF, all the four samples were characterized before and after coating and their properties were analyzed. Characteristics of four different sample fabrics before and after coating are presented in Table-9 and properties of four different sample fabrics before and after coating are shown in Table-10.

Table 9. Characteristics of PTFE membrane laminated samples before and after coating

Fabric sample code	Areal density of uncoated fabric (g/m^2)		Areal density of coated fabric (g/m^2)		Increase in areal density after coating (%)	Thickness of uncoated fabric (mm)		Thickness of coated fabric (mm)		Density of uncoated fabric (Kg/m^3)		Density of coated fabric (Kg/m^3)	
	Mean	SD	Mean	SD		Mean	SD	Mean	SD	Mean	SD	Mean	SD
WMK-1	89	2.24	99	2.31	11.24	0.21	0.01	0.23	0.01	423.81	2.21	430.43	4.85
WMK-2	167	2.29	185	2.19	10.78	0.35	0.01	0.38	0.01	477.14	2.20	486.84	3.15
WMF-3	314	1.62	339	3.74	7.96	1.20	0.01	1.25	0.02	261.67	1.45	271.20	2.01
WMF-4	389	1.22	418	3.56	7.46	1.27	0.01	1.32	0.02	306.30	2.25	316.67	1.74

Table 10. Properties of PTFE membrane laminated samples before and after coating

Properties		Uncoated				Coated			
		WMK-1	WMK-2	WMF-3	WMF-4	WMK-1	WMK-2	WMF-3	WMF-4
Hydrostatic resistance (cmH_2O)	Mean	1464	1522	1087	1114	1616	1712	1192	1216
	SD	6.55	6.13	4.55	4.11	7.79	7.36	6.48	6.24
Breaking force (N)	Mean	410	478	356	379	477	572	412	435
	SD	5.50	7.69	7.94	9.44	8.47	8.36	9.58	10.38
Bending rigidity (10^{-6} Nm)	Mean	7.56	13.58	27.19	28.10	8.01	14.63	28.62	29.53
	SD	0.17	0.12	0.15	0.12	0.18	0.13	0.16	0.12
Evaporative resistance, R_{et} (m^2Pa / W)	Mean	6.44	8.42	11.64	12.76	6.74	8.84	11.92	13.00
	SD	0.21	0.27	0.22	0.21	0.29	0.31	0.25	0.28
RWVP (%)	Mean	47.60	42.74	33.58	30.64	47.28	42.40	33.26	30.30
	SD	0.37	0.23	0.28	0.29	0.41	0.36	0.33	0.30
Air permeability ($l/m^2/s$)	Mean	1.07	0.72	1.51	1.32	1.04	0.68	1.45	1.26
	SD	0.04	0.05	0.06	0.07	0.04	0.05	0.07	0.07

3.2.2.2 Spray test

To determine the resistance of a fabric to wetting by water, spray test method is used. It is usually used to determine the water-repellent effect of finishes applied to fabrics. Pro-ser Spray Rating Tester was used in the experiment for conducting spray test according to AATCC 22 [68]. The coated samples were conditioned at 21°±1°C for 24 hours under a relative humidity of 65±2% before testing. The samples were stretched on a hoop which was held at an angle of 45° and 250 mL water was poured through a spray nozzle. Any wetting or spotted pattern was observed and compared with the photographic rating chart as below [69].

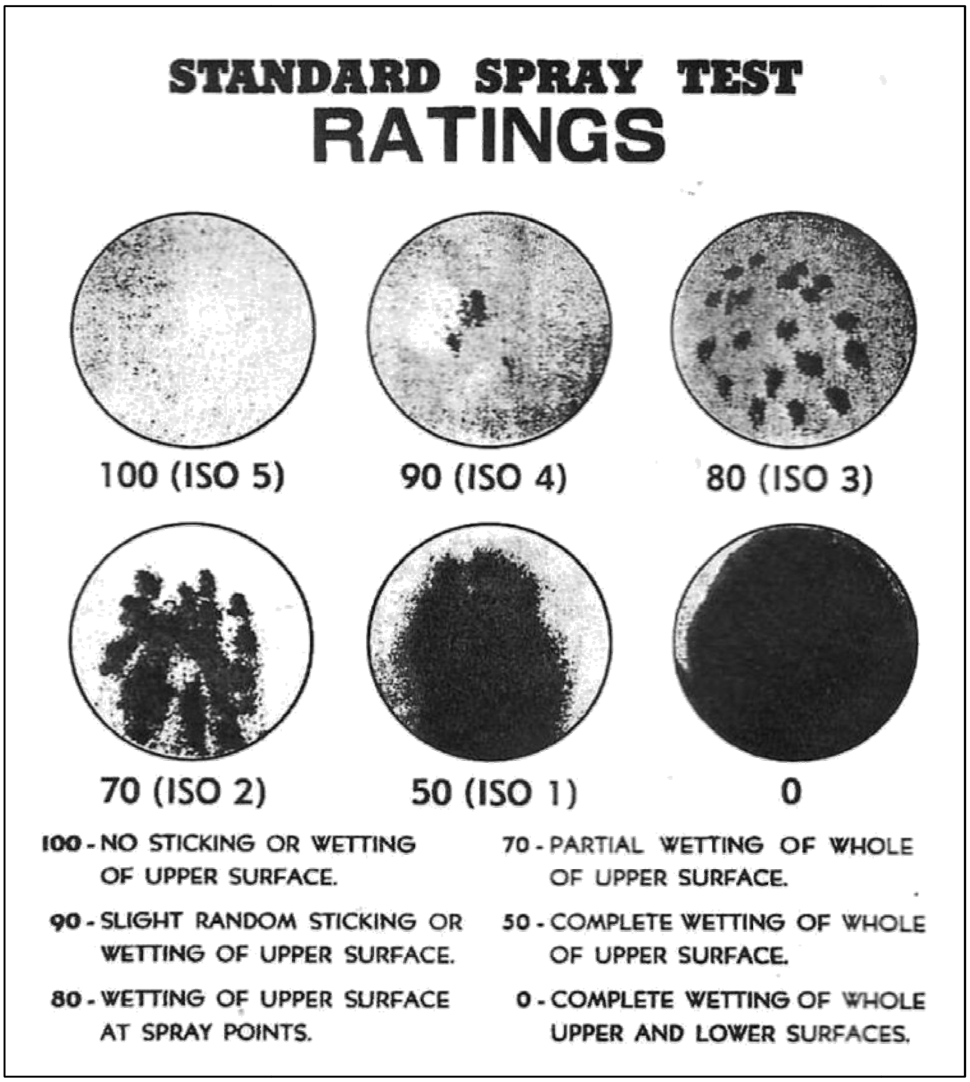

Figure 15. Spray test rating chart.

3.2.2.3 Analysis of variance (ANOVA)

Analysis of variance (ANOVA) was used to determine the statistical significance of variations. To deduce whether the variations of different properties before and after coating for the samples were either significant or not, P-value was examined. It was considered as a statistical significant change in the result if P-value was found less than 0.05 ($p < 0.05$), otherwise, there was no significant change.

CHAPTER 4: RESULTS AND DISCUSSION

This chapter describes the results and discussion of the two parts of the experiment like the followings:

4.1 Results and discussion for evaluation of different test results of PU membrane laminated fabrics

4.1.1 Morphological cross-section

Figure-16 shows the images of two-layered and three-layered samples obtained from scanning electron microscope (SEM). It gives a proper idea about the morphological cross-sections of six different types of PU membrane laminated sample fabrics prepared in the experiment. From the images, it is evident that FM-1, FM-2 and FM-3 are two-layered fabrics, where upper layers of all these three samples are outer polyester plain woven fabrics and lower layers are polyurethane membranes. On the other hand, FMK-4, FMK-5 and FMK-6 samples from the images are three-layered laminated fabrics. Here, upper layers of these samples are polyester plain woven fabrics, middle layers are polyurethane membranes and lower layers of these sample fabrics are inner polyester knitted fabrics.

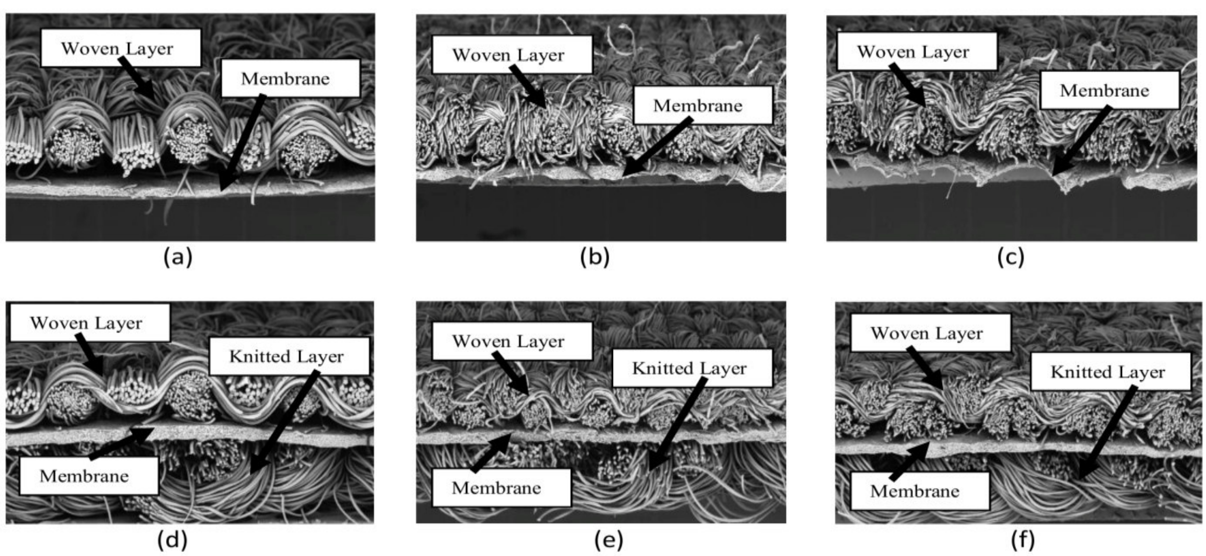

Figure 16. SEM images (x 200) of two-layered samples: (a) FM-1 (b) FM-2 (c) FM-3 and three-layered samples: (d) FMK-4 (e) FMK-5 (f) FMK-6.

4.1.2 Analysis of hydrostatic resistance

Breathability is meaningless without proper hydrostatic resistance. It is evident from the test results of hydrostatic resistance (Table-5), that all PU membrane laminated sample fabrics prepared in the experiment show the values more than 500 cmH_2O and these fabrics can be used as outdoor sports clothing. However, P-value from ANOVA in Table-11 for fabric weight and hydrostatic resistance is found less than 0.05 which explains a significant influence of fabric weight on hydrostatic resistance of the samples. Pearson correlation coefficient (r) for fabric weight and hydrostatic resistance is obtained +0.9743 from Table-12 that represents a strong positive correlation between fabric weight and hydrostatic resistance of the sample fabrics. Coefficient of determination (R^2) value is 0.9493 that denotes a good strength linear association between fabric weight and hydrostatic resistance of different samples. Here, P-value, r-value and R^2-value express that hydrostatic resistance of the sample is significantly increased when fabric weight of the sample is increased. Again, P-value is obtained less than 0.05 in ANOVA Table-13 for fabric density and hydrostatic resistance which also explains a significant influence of fabric density on hydrostatic resistance of the samples. Here, r-value and R^2-value are obtained +0.9988 and 0.9976 respectively from Table-14. This also indicates a strong positive relation between fabric density and hydrostatic resistance. If two-layered and three-layered samples are considered separately, then R^2-value of three-layered samples is little bit higher than R^2-value of two-layered samples in case of relation between fabric weight and hydrostatic resistance from Figure-18. But, R^2-value is little bit higher for two-layered samples than R^2-value of three-layered samples in case of relation between fabric density and hydrostatic resistance from Figure-20.

However, all three-layered samples show better hydrostatic resistance property than all two-layered samples. Because there are increases of fabric weight and fabric density for all three-layered samples when knitted fabrics are added as inner layers during their lamination process resulting in the increases of hydrostatic resistance values (Figure-17, 18, 19 & 20). Among the six samples, highest hydrostatic resistance property is obtained for FMK-4 sample due to its highest fabric weight and fabric density and lowest is found for FM-3 sample due to its lowest fabric weight and fabric density. When compared only two-layered three samples to each other, the best hydrostatic resistance is obtained for FM-1 sample and when compared only three-layered three samples to each other, the highest hydrostatic resistance is found for FMK-4 sample. The reason is that outer woven layer parts of FM-1 and FMK-4 samples are prepared by F-1 polyester woven fabric whose warp cover factor and weft cover factor are more than warp and weft cover factors of F-2 and F-3 polyester woven fabrics. And more warp and weft cover factors of outer layers of these two samples influence in resulting more hydrostatic resistance property.

Table 11. ANOVA for fabric weight and hydrostatic resistance

	df	SS	MS	F	P-value
Regression	1	35349.62	35349.62	74.87812	0.000981
Residual	4	1888.382	472.0954		
Total	5	37238			

Table 12. Correlation between fabric weight and hydrostatic resistance

Correlation equation	HR = 1.232FW + 457.67 HR = Hydrostatic resistance FW = Fabric weight
Pearson correlation coefficient (r)	+ 0.9743
Coefficient of determination (R^2)	0.9493

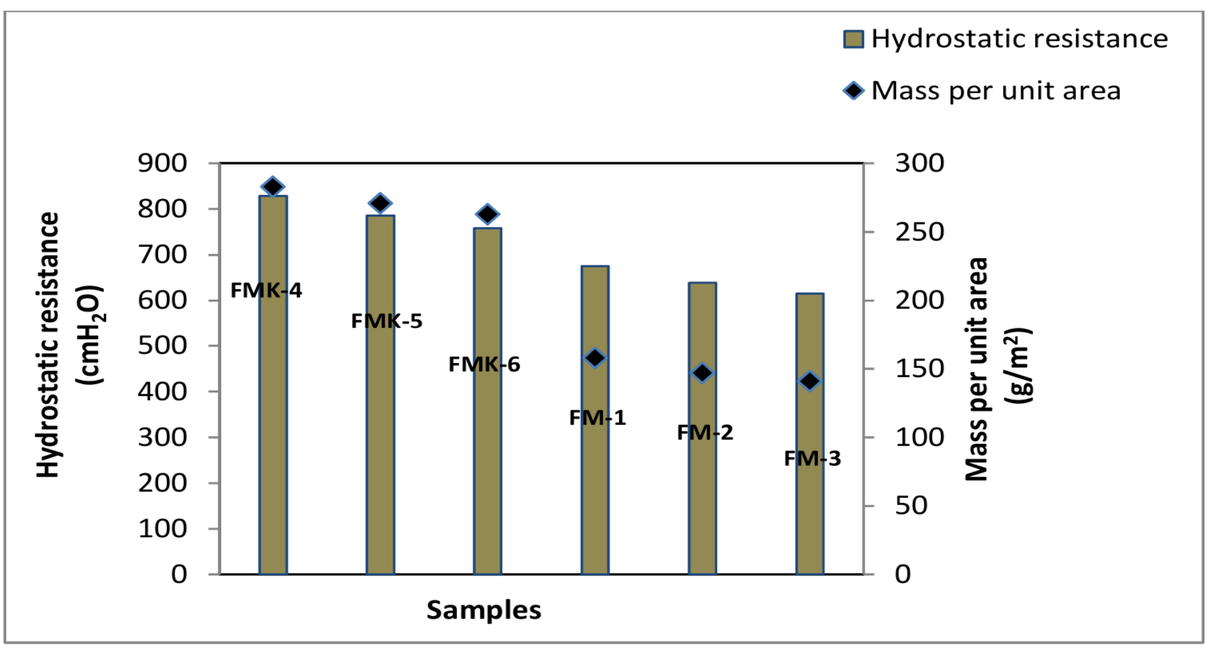

Figure 17. Fabric weight vs. hydrostatic resistance.

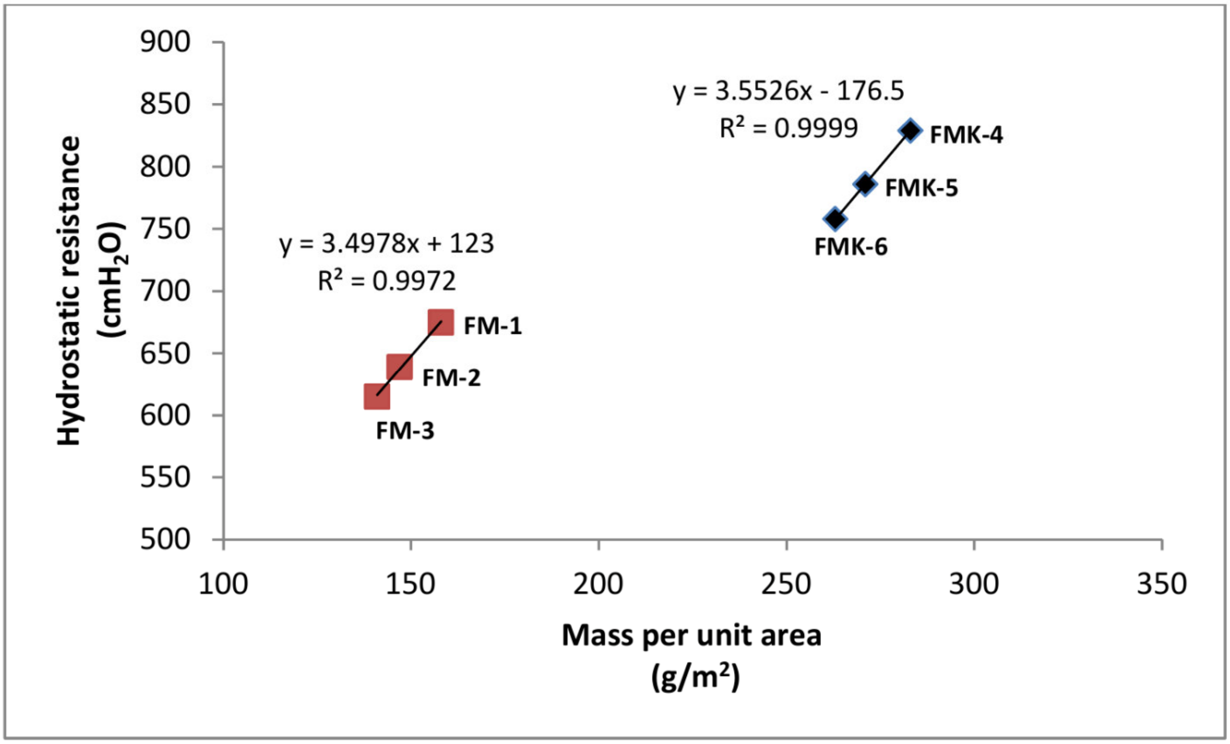

Figure 18. Effect of fabric weight on hydrostatic resistance for two-layered and three-layered samples.

Table 13. ANOVA for fabric density and hydrostatic resistance

	df	SS	MS	F	P-value
Regression	1	37147.21	37147.21	1636.664	2.23E-06
Residual	4	90.78766	22.69691		
Total	5	37238			

Table 14. Correlation between fabric density and hydrostatic resistance

Correlation equation	HR = 4.5371FD - 1027.1 HR = Hydrostatic resistance FD = Fabric density
Pearson correlation coefficient (r)	+ 0.9988
Coefficient of determination (R^2)	0.9976

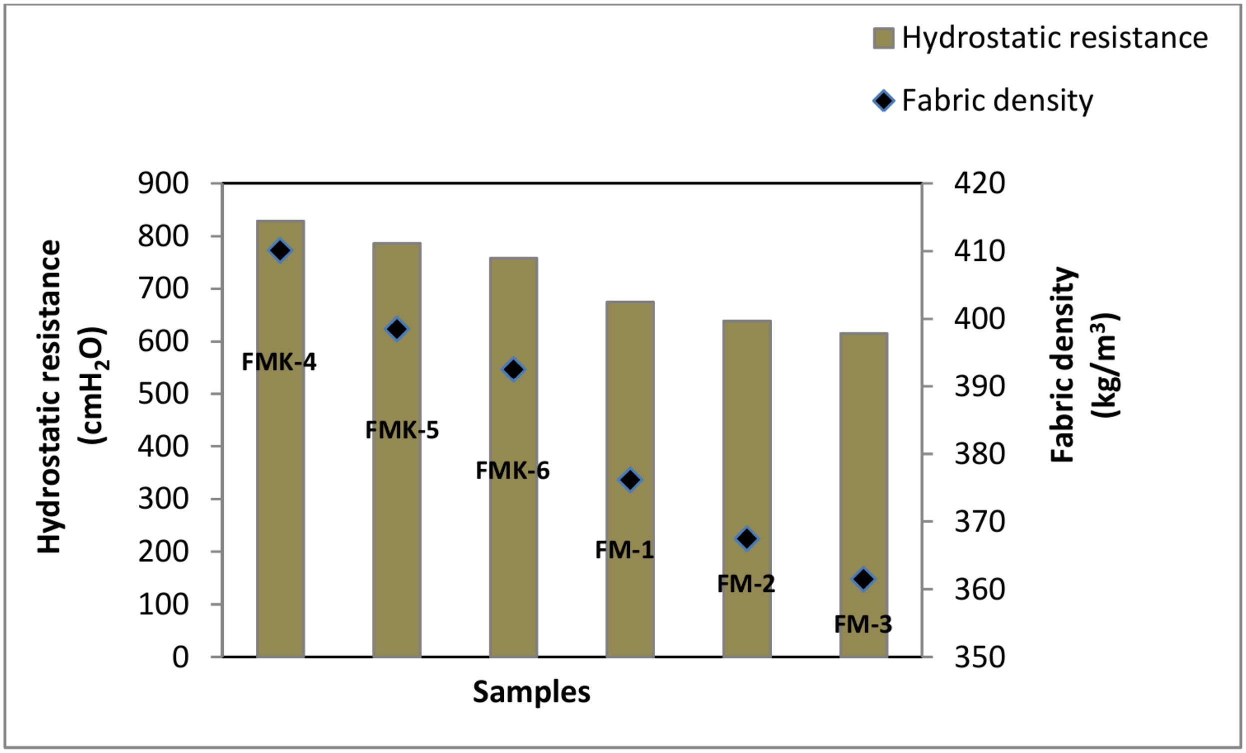

Figure 19. Fabric density vs. hydrostatic resistance.

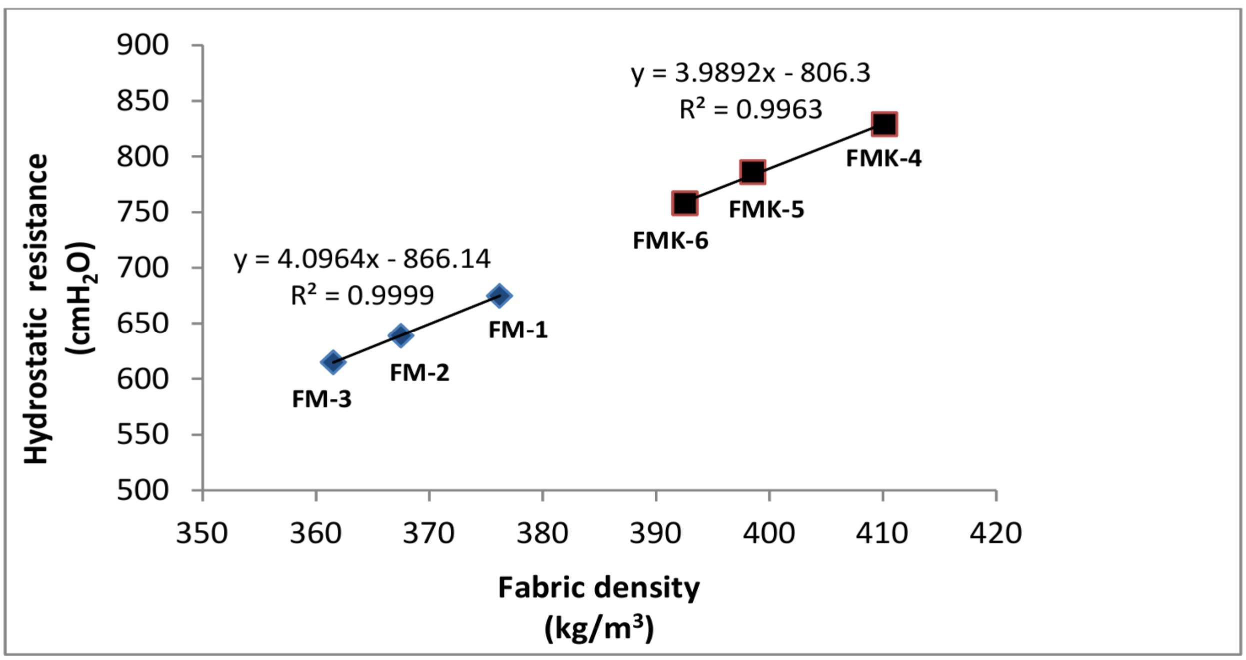

Figure 20. Effect of fabric density on hydrostatic resistance for two-layered and three-layered samples.

4.1.3 Analysis of water vapour permeability

Water vapour permeability of a fabric is obtained by measuring relative water vapour permeability (RWVP) and evaporative resistance (R_{et}). Increased RWVP and decreased R_{et} values determine the higher water vapour transmission of the measuring samples. R_{et} represents the water vapour pressure difference between the two sides of the specimen divided by the resultant evaporative heat flux per unit area in the direction of the gradient as the unit of m^2Pa/W. The obtained R_{et} values of all prepared PU membrane laminated samples are between 6-10 m^2Pa/W (Table-5). This means that all the prepared samples are good breathable fabrics. From ANOVA (Table-15) of fabric weight and RWVP, P-value is less than 0.05 and from ANOVA (Table-19) of fabric weight and evaporative resistance, P-value is less than 0.05. This indicates that there are significant influences of fabric weights of the sample fabrics on their RWVP and evaporative resistance. But, from Table-16, r-value and R^2-value are -0.9531 and 0.9084 respectively when correlation between fabric weight and RWVP is considered. This represents a negative strong relationship between fabric weight and RWVP as well as denotes a good linear relationship between them. On the other hand, r-value is +0.9539 and R^2-value is 0.91 from Table-20 when correlation between fabric weight and evaporative resistance is considered. This represents a positive strong linear relationship between fabric weight and evaporative resistance. Laminated sample fabric becomes more comfortable when the fabric weight is lower, as there is an increase of RWVP or a decrease of evaporative resistance with the decrease of fabric weight.

Again, from ANOVA of Table-17, P-value is obtained less than 0.05 that indicates the significant influence of fabric thickness on RWVP and from ANOVA of Table-21, P-value is obtained less than 0.05 that indicates the significant influence of fabric thickness on evaporative resistance. From Table-18, r-value and R^2-value are found -0.9373 and 0.8785 respectively and this indicates a good negative relationship between fabric thickness and RWVP. But, from Table-22, r-value is +0.9388 and R^2-value is 0.8815. This expresses a positive good relationship between fabric thickness and evaporative resistance. Here also laminated sample fabric becomes more comfortable with the decrease of fabric thickness, as there is an increase of RWVP and a decrease of evaporative resistance with the decrease of fabric thickness. However, fabric weight has more influence than fabric thickness on RWVP and evaporative resistance for the sample fabrics. Because, r-values and R^2-values from correlation between fabric weight with RWVP and evaporative resistance are more than r-values and R^2-values from correlation between fabric thickness with RWVP and evaporative resistance.

Table 15. ANOVA for fabric weight and relative water vapour permeability

	df	SS	MS	F	P-value
Regression	1	59.45464	59.45464	39.6781	0.003246
Residual	4	5.993698	1.498425		
Total	5	65.44833			

Table 16. Correlation between fabric weight and relative water vapour permeability

Correlation equation	RWVP = -0.0505FW + 54.759 RWVP = Relative water vapour permeability FW = Fabric weight
Pearson correlation coefficient (r)	- 0.9531
Coefficient of determination (R^2)	0.9084

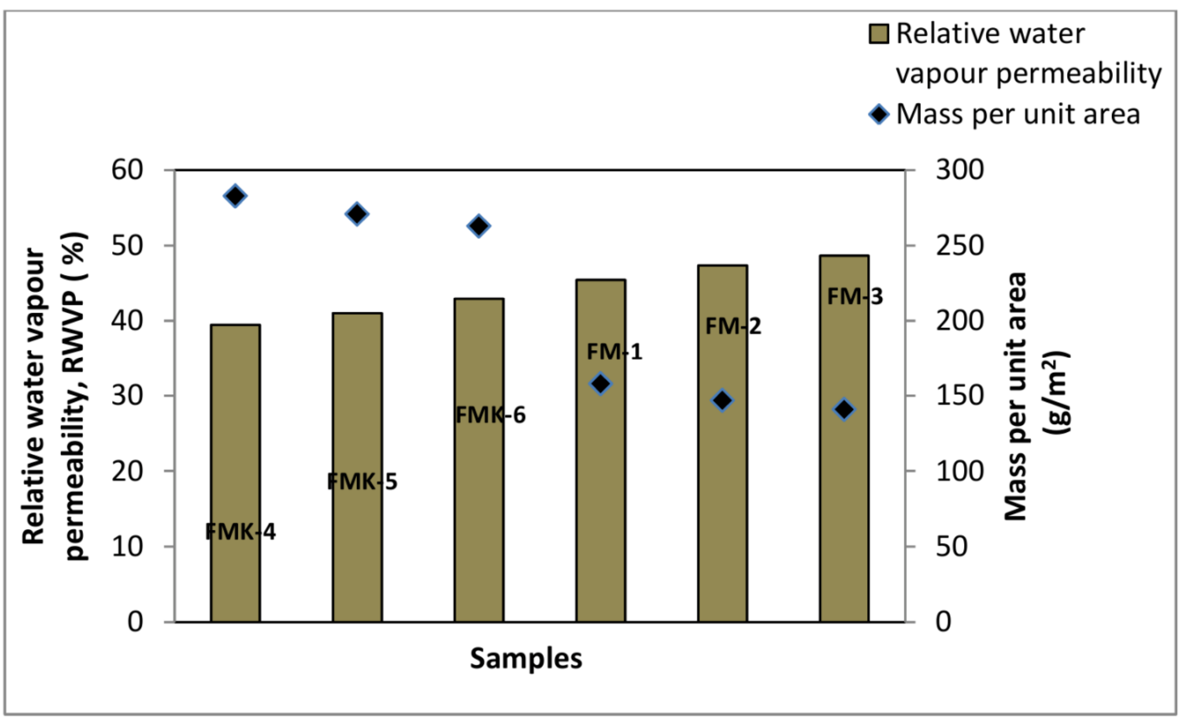

Figure 21. Fabric weight vs. relative water vapour permeability.

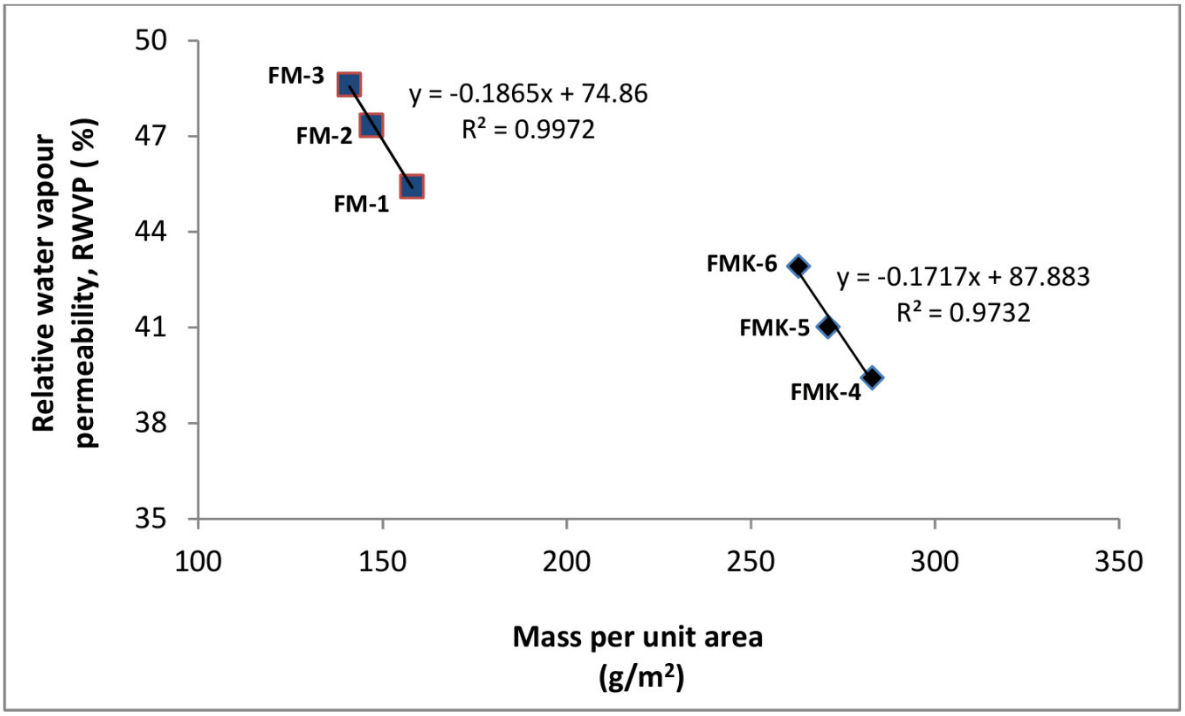

Figure 22. Effect of fabric weight on relative water vapour permeability for two-layered and three-layered samples.

Table 17. ANOVA for fabric thickness and relative water vapour permeability

	df	SS	MS	F	P-value
Regression	1	57.49808	57.49808	28.92893	0.005774
Residual	4	7.950253	1.987563		
Total	5	65.44833			

Table 18. Correlation between fabric thickness and relative water vapour permeability

Correlation equation	RWVP = -22.313FT + 56.21 RWVP = Relative water vapour permeability FT = Fabric thickness
Pearson correlation coefficient (r)	- 0.9373
Coefficient of determination (R^2)	0.8785

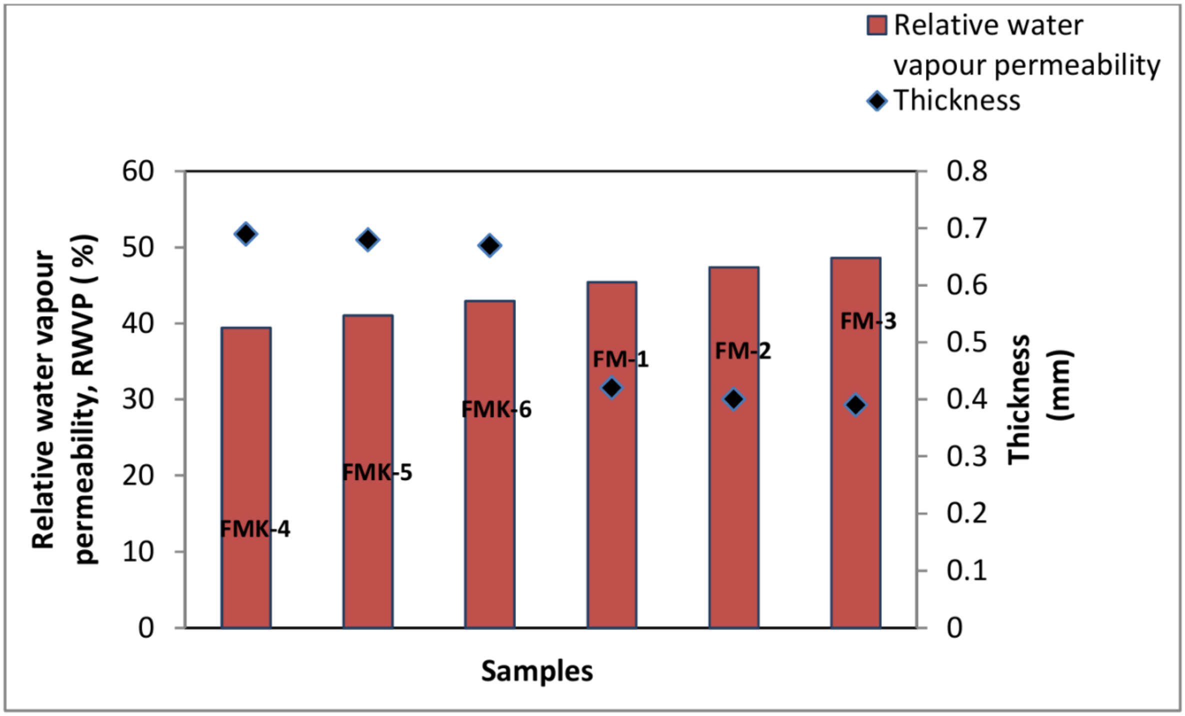

Figure 23. Fabric thickness vs. relative water vapour permeability.

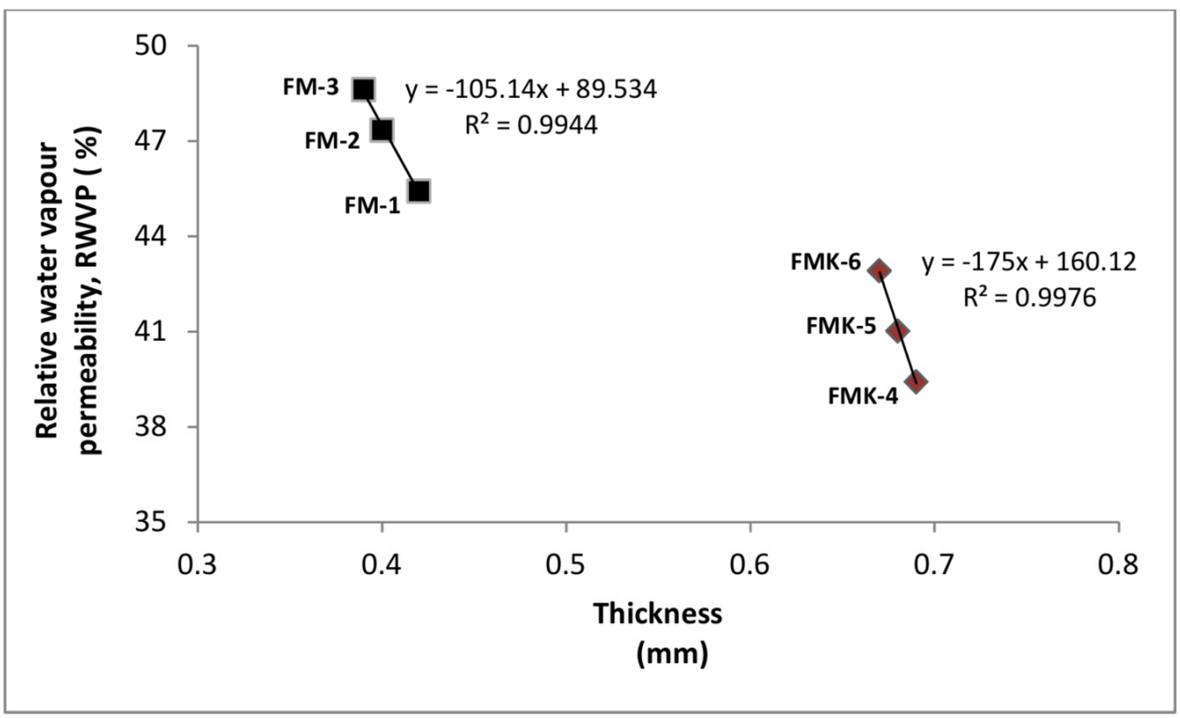

Figure 24. Effect of fabric thickness on relative water vapour permeability for two-layered and three-layered samples.

Table 19. ANOVA for fabric weight and evaporative resistance

	df	SS	MS	F	P-value
Regression	1	5.373343	5.373343	40.45756	0.003132
Residual	4	0.531257	0.132814		
Total	5	5.9046			

Table 20. Correlation between fabric weight and evaporative resistance

Correlation equation	ER = 0.0152FW + 4.5928 ER = Evaporative resistance FW = Fabric weight
Pearson correlation coefficient (r)	+ 0.9539
Coefficient of determination (R^2)	0.91

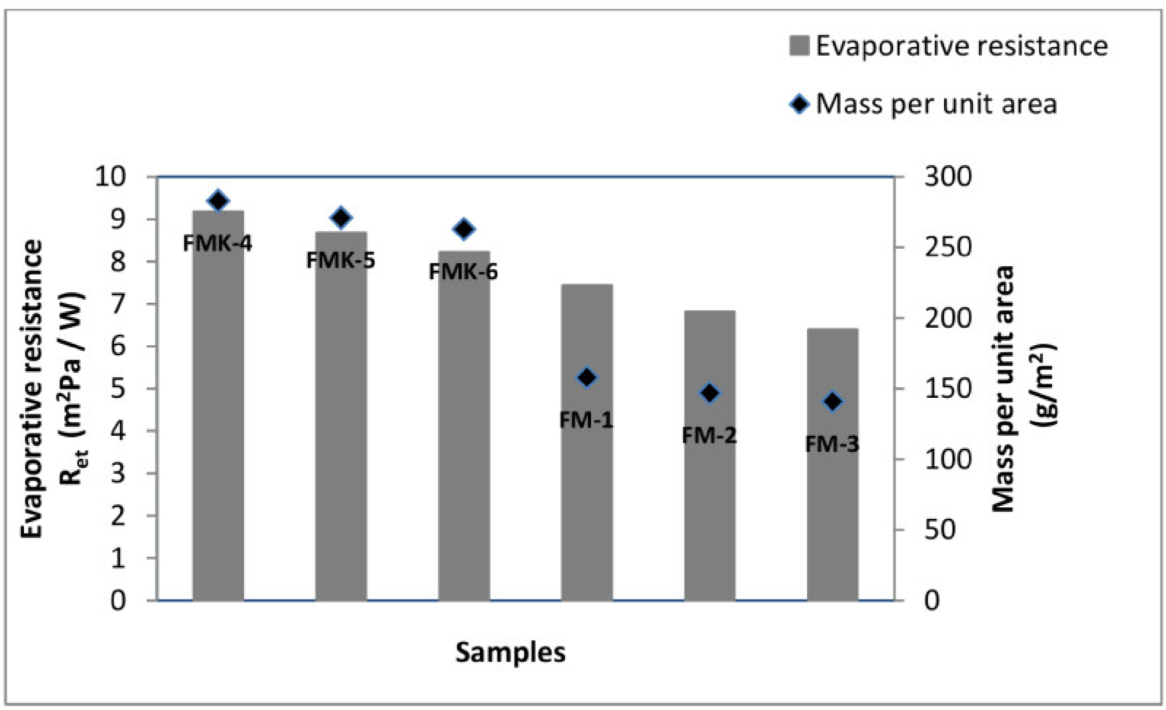

Figure 25. Fabric weight vs. evaporative resistance.

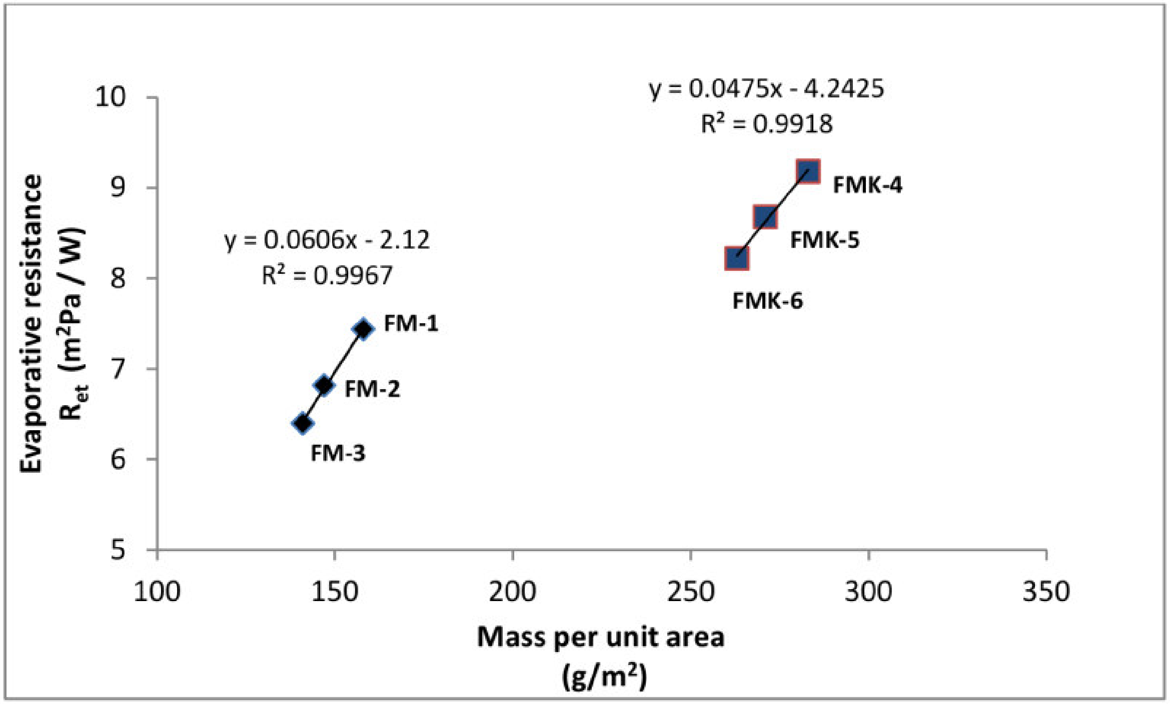

Figure 26. Effect of fabric weight on evaporative resistance for two-layered and three-layered samples.

Table 21. ANOVA for fabric thickness and evaporative resistance

	df	SS	MS	F	P-value
Regression	1	5.204994	5.204994	29.75959	0.005487
Residual	4	0.699606	0.174901		
Total	5	5.9046			

Table 22. Correlation between fabric thickness and evaporative resistance

Correlation equation	ER = 6.7135FT + 4.1535 ER = Evaporative resistance FT = Fabric thickness
Pearson correlation coefficient (r)	+ 0.9388
Coefficient of determination (R^2)	0.8815

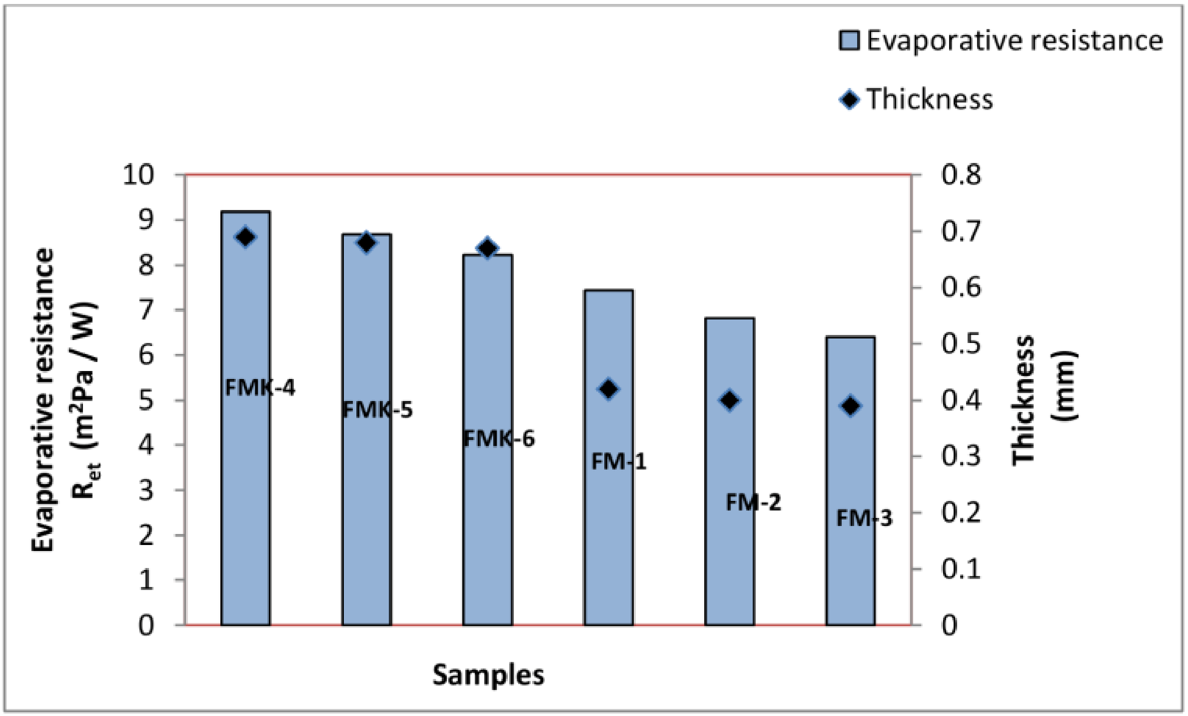

Figure 27. Fabric thickness vs. evaporative resistance.

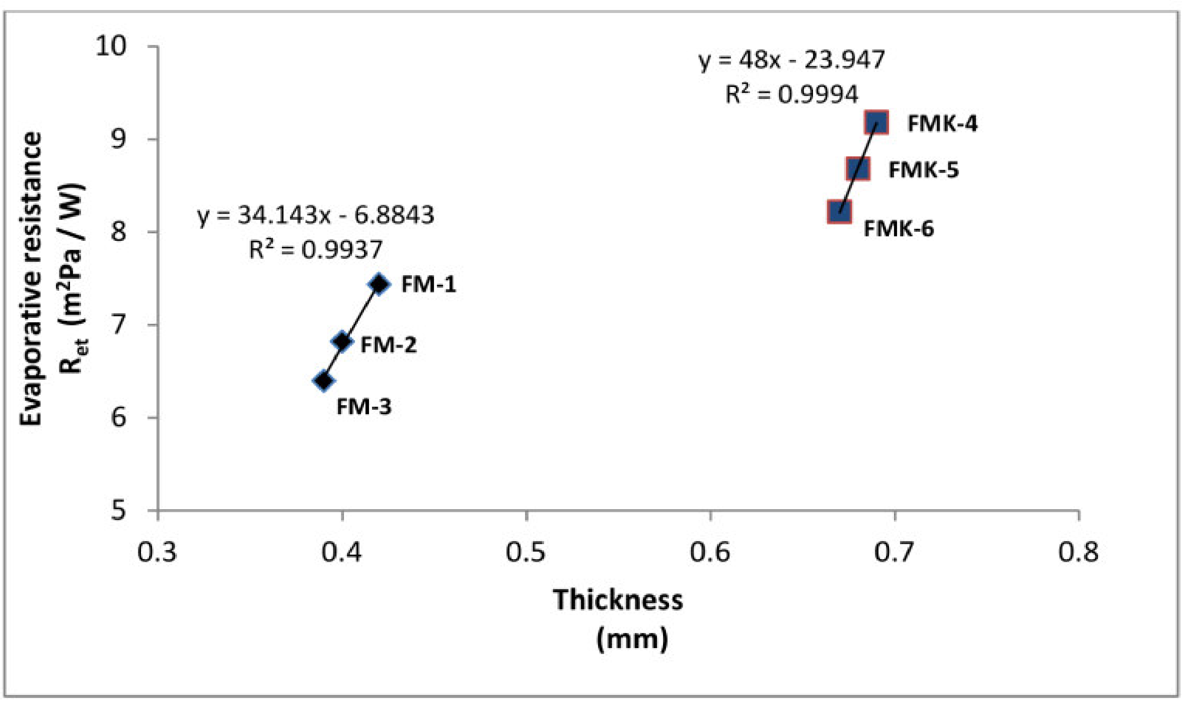

Figure 28. Effect of fabric thickness on evaporative resistance for two-layered and three-layered samples.

From the Figures (21, 22, 23 & 24), it is evident that relative water vapour permeability of the two-layered sample fabrics are higher than relative water vapour permeability of the three-layered fabrics. And from the Figures (25, 26, 27 & 28), it is clear that evaporative resistance of three-layered samples are higher than evaporative resistance of two-layered samples. Because, when polyester knitted fabrics are added as inner layers in all three-layered samples, then fabric weight and fabric thickness of these samples are also increased resulting in less relative water vapour permeability and more evaporative resistance during comparison with two-layered samples. However, among all the samples, highest water vapour permeability is obtained in case of sample FM-3 with lowest fabric weight and thickness. This sample is also prepared with F-3 polyester woven fabric as an outer layer whose fabric weight is also lower than the fabric weights of F-1 and F-2 polyester woven fabrics due to its lower warp and weft cover factor. On the other hand, lowest water vapour permeability is obtained for the sample FMK-4 due to its highest fabric weight and thickness among all fabrics. Moreover, this sample is produced by F-1 polyester woven fabric as an outer layer whose weight is higher than the weights of F-2 and F-3 polyester woven fabrics due to its higher warp and weft cover factor.

4.1.4 Analysis of air permeability

From ANOVA of Table-23, P-value is obtained less than 0.05 which expresses that there is a significant influence of fabric thickness on air permeability for different types of laminated sample fabrics. From Table-24, r-value is -0.8639 and R^2-value is 0.7464 which indicates that there is a good negative linear relationship between fabric thickness of the laminated samples and their air permeability. Again, from ANOVA of Table-25, P-value is found less than 0.05 which also expresses that there is a significant influence of fabric density on air permeability for the laminated sample fabrics. From Table-26, r-value is -0.9703 and R^2-value is 0.9415 which indicates that there is a strong negative linear relationship between fabric density of the samples and their air permeability. From this analysis, it is also clear that the influence of fabric density on air permeability of different samples is more than the influence of fabric thickness on air permeability. Because, r-value and R^2-value obtained from correlation between fabric density and air permeability are more than r-value and R^2-value obtained from correlation between fabric thickness and air permeability.

Table 23. ANOVA for fabric thickness and air permeability

	df	SS	MS	F	P-value
Regression	1	0.690085	0.690085	11.7704	0.0265161
Residual	4	0.234515	0.058629		
Total	5	0.9246			

Table 24. Correlation between fabric thickness and air permeability

Correlation equation	AP = -2.4445FT + 3.3141 AP = Air permeability FT = Fabric thickness
Pearson correlation coefficient (r)	- 0.8639
Coefficient of determination (R^2)	0.7464

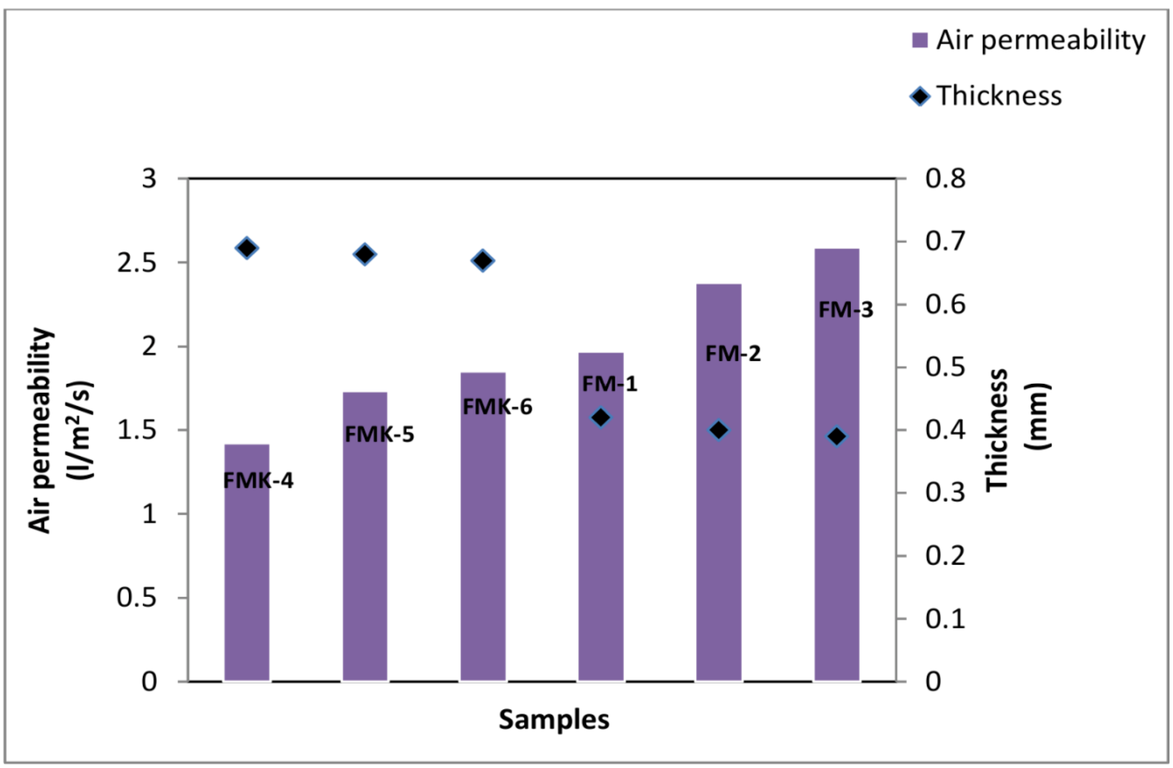

Figure 29. Fabric thickness vs. air permeability.

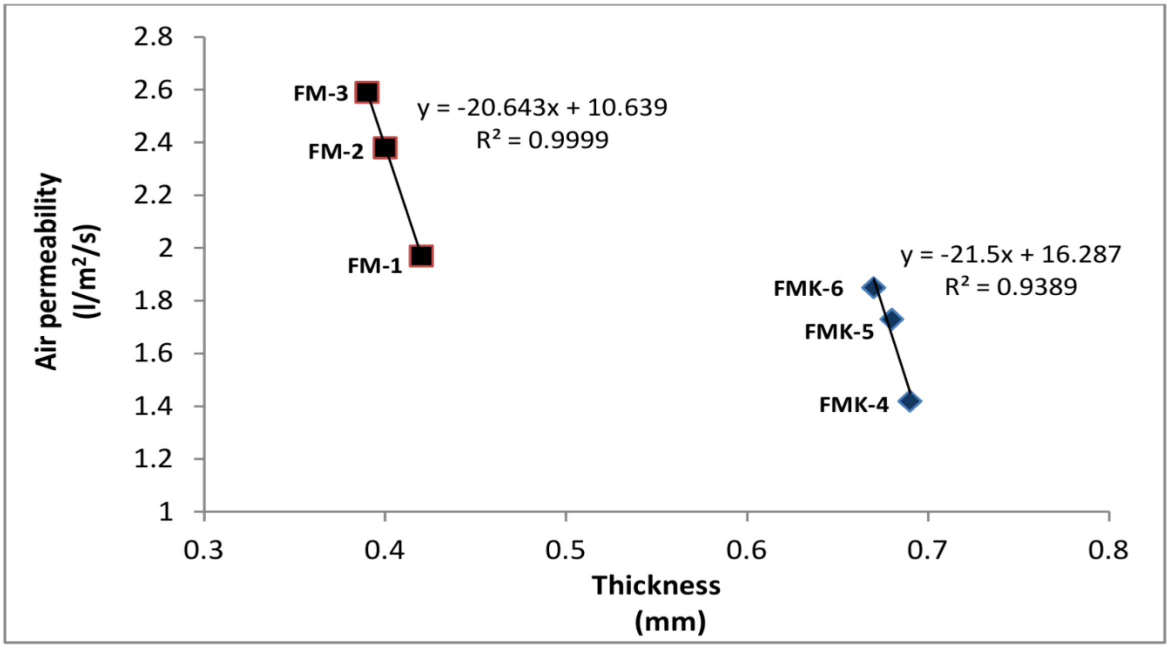

Figure 30. Effect of fabric thickness on air permeability for two-layered and three-layered samples.

Table 25. ANOVA for fabric density and air permeability

	df	SS	MS	F	P-value
Regression	1	0.870513	0.870513	64.37889	0.001309
Residual	4	0.054087	0.013522		
Total	5	0.9246			

Table 26. Correlation between fabric density and air permeability

Correlation equation	AP = -0.022FD + 10.433 AP = Air permeability FD = Fabric density
Pearson correlation coefficient (r)	- 0.9703
Coefficient of determination (R^2)	0.9415

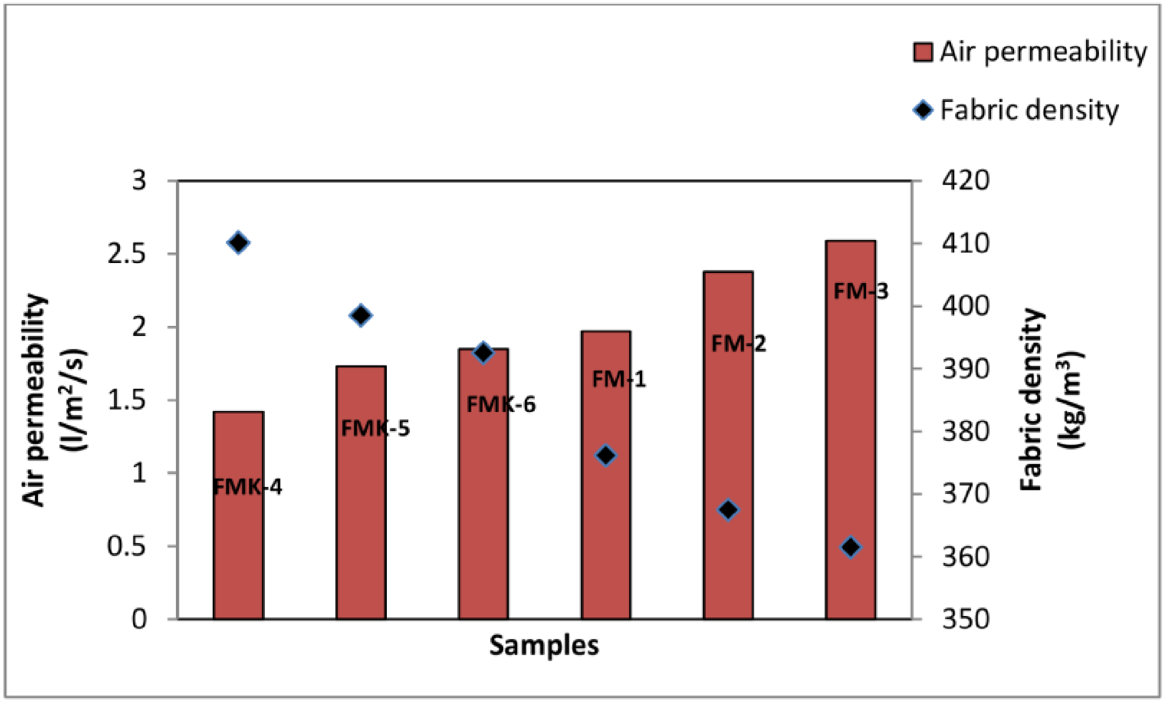

Figure 31. Fabric density vs. air permeability.

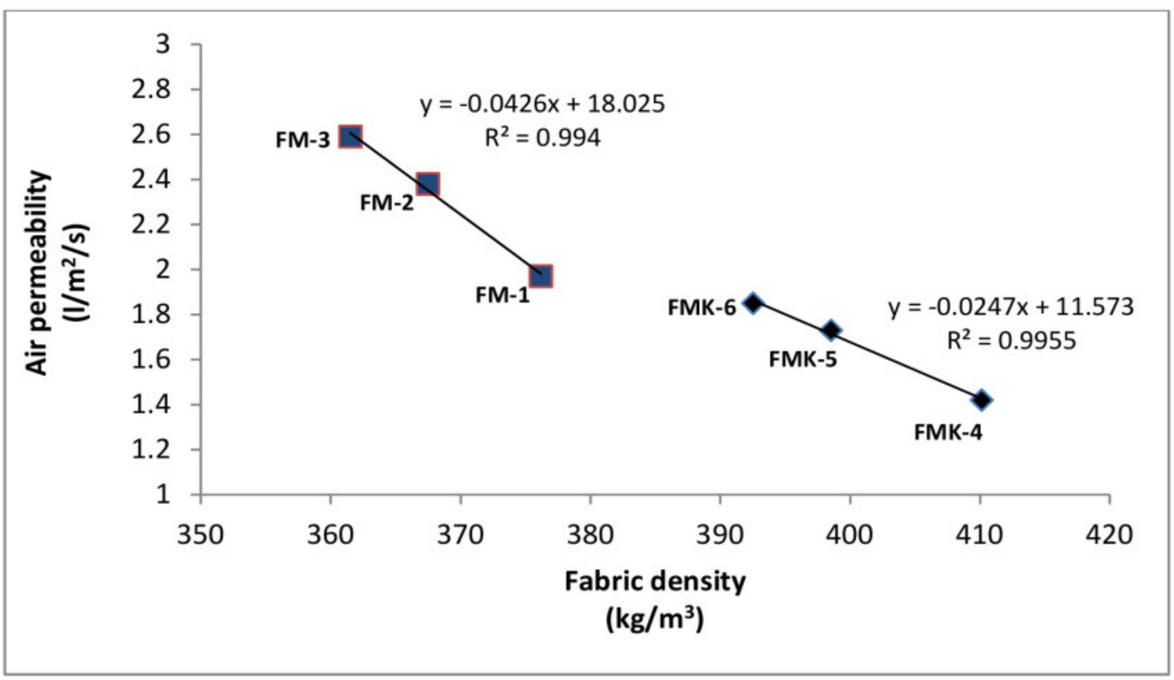

Figure 32. Effect of fabric density on air permeability for two-layered and three-layered samples.

Among all the samples, two-layered samples have more air permeability than three-layered samples. The reason is when knitted layer is added as an inner layer for each of three-layered samples then, both fabric thickness and fabric density also increase resulting in less air permeability than two-layered laminated sample fabrics. Moreover, due to use of knitted layer for each three-layered sample, there is more air entrapment that also causes less air permeability. So, fabric air permeability with membrane is attributed here due to higher compactness and air entrapment that offer resistance to the passage of air. From the Figures (29, 30, 31 & 32), it can be observed that FM-3 sample has the highest air permeability value among all the samples for its lowest fabric thickness and density. Moreover, its outer woven layer part is F-3 polyester woven fabric whose warp cover factor and weft cover factor are lower than the cover factors of F-1 and F-2 polyester woven fabrics. On the other hand, lowest air permeability is obtained for FMK-4 sample due to its highest fabric thickness and density as well as its outer woven layer part is F-1 polyester woven fabric whose warp cover factor and weft cover factor are more than the cover factors of F-2 and F-3 polyester woven fabrics. Outer layer parts of two-layered FM-1 sample and three-layered FMK-4 sample are prepared with F-1 polyester woven fabric, but air permeability value for FM-1 is higher than FMK-4 due to its lower fabric thickness and density.

4.1.5 Analysis of thermal resistance

Thermal resistance can be defined as a measure of the body's ability to prevent heat from flowing through it. Under a certain condition of climate, if the thermal resistance of clothing is small, the heat energy will gradually reduce with a sense of coolness [70]. Thermal conductivity and thermal resistance are opposite to each other. If thermal resistance of a fabric increases, thermal conductivity decreases. For ideal conditions, thermal resistance, $R = h / \lambda$, where h is thickness and λ is thermal conductivity of the fabric. However, from ANOVA of Table-27, P-value is less than 0.05 that clarifies a significant influence of thickness of the laminated layered samples on their thermal resistance. Again, Pearson correlation coefficient (r) is +0.9459 and coefficient of determination (R^2) is 0.8947 from Table-28. This indicates a good positive linear relationship between fabric thickness and thermal resistance property of the samples. It means that thermal resistance of the sample fabric increases with the increase of fabric thickness.

Table 27. ANOVA for fabric thickness and thermal resistance

	df	SS	MS	F	P-value
Regression	1	47.89675	47.89675	33.9996	0.00431
Residual	4	5.634979	1.408745		
Total	5	53.53173			

Table 28. Correlation between fabric thickness and thermal resistance

Correlation equation	TR = 20.365FT + 4.0421 TR = Thermal resistance FT = Fabric thickness
Pearson correlation coefficient (r)	+ 0.9459
Coefficient of determination (R^2)	0.8947

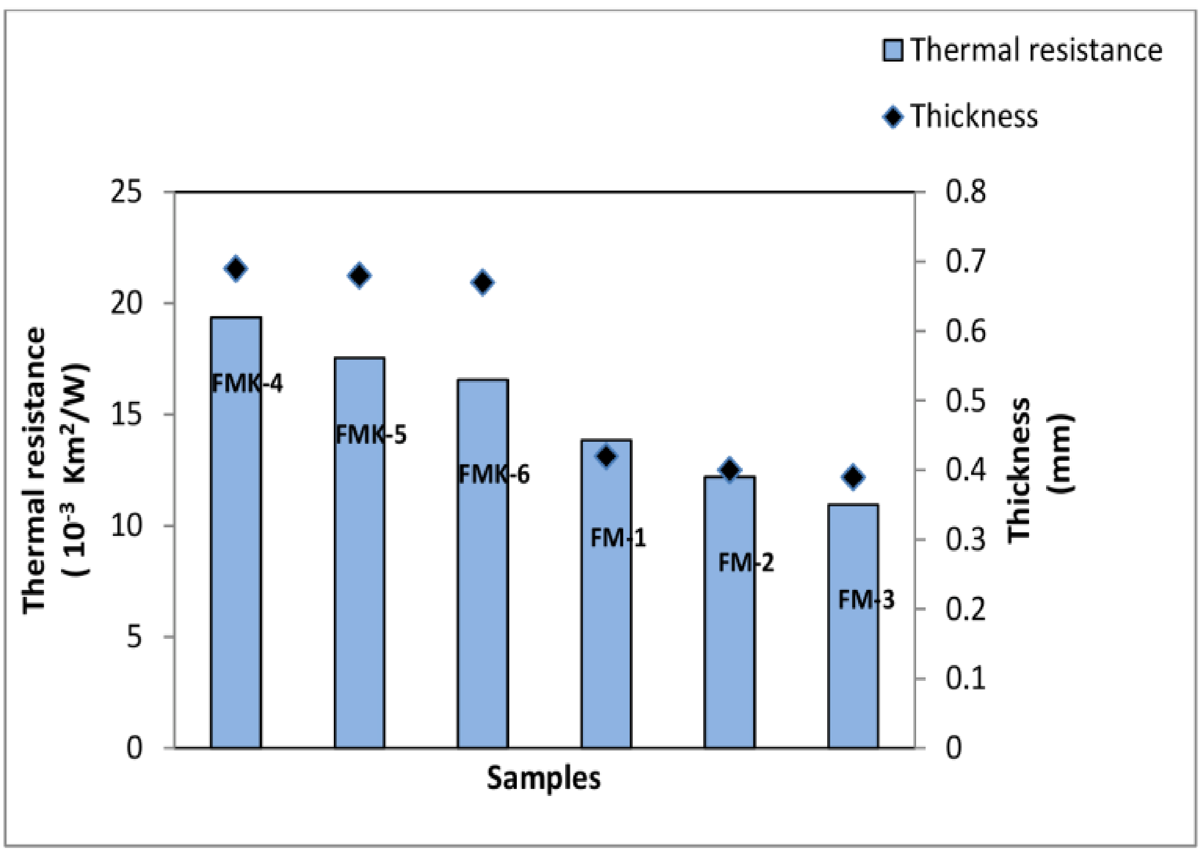

Figure 33. Fabric thickness vs. thermal resistance.

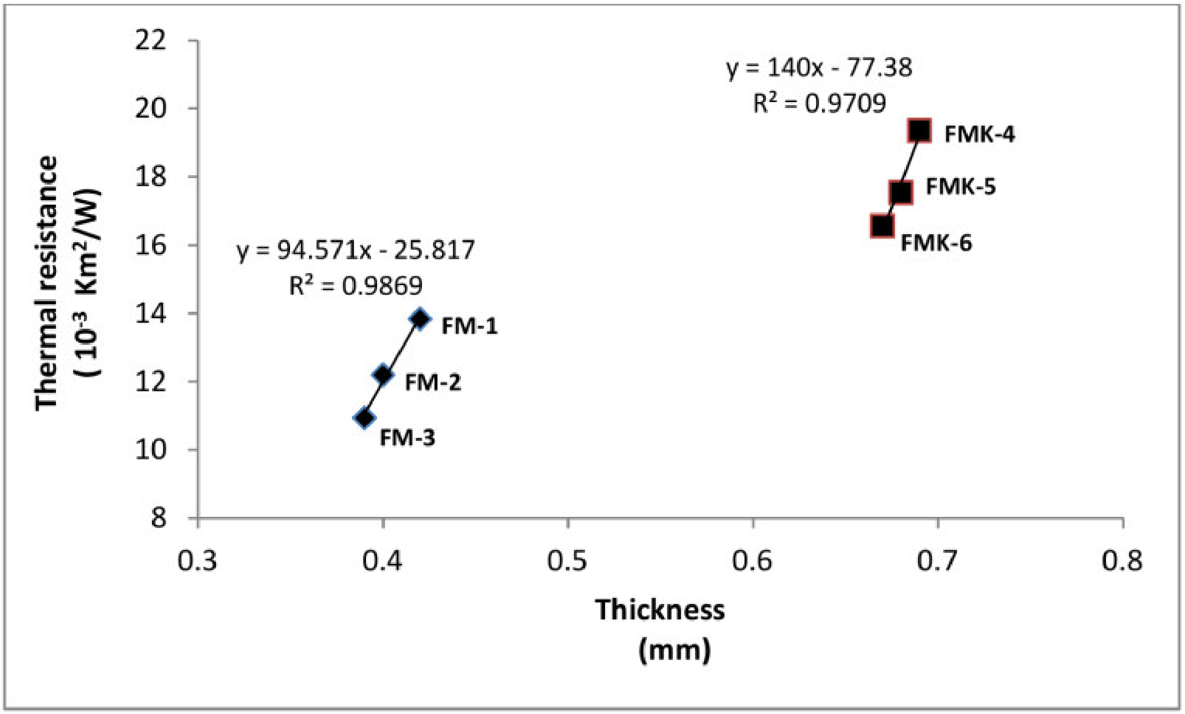

Figure 34. Effect of fabric thickness on thermal resistance for two-layered and three-layered samples.

Among all the six samples, highest thermal resistance is obtained for FMK-4 sample due to its highest thickness value and lowest thermal resistance value is obtained in case of sample FM-3 for its lowest thickness value obtained from Figure-33 and Figure-34. All three-layered samples have more thermal resistance than all two-layered samples. Because when knitted fabric is added during lamination process, thickness of the three-layered fabric also increases. Moreover, due to knitted part, there are more air gaps between fibers which decrease the heat transfer with increasing thermal resistance. So, these three-layered fabrics with better thermal resistance are more suitable than two-layered fabrics in comparatively cold condition.

4.1.6 Analysis of bending rigidity

Bending rigidity represents the fabric stiffness property. Very stiff fabric can be uncomfortable and unfit for use. It is evident from Figure-35 and Figure-36 that thickness is the determining factor which influences on bending rigidity of the layered laminated fabric samples. Here, Pearson correlation coefficient (r) is +0.9999 and Coefficient of determination (R^2) is 0.9999 obtained from Table-30 and P-value from ANOVA of Table-29 is less than 0.05 that clearly determines a strong positive significant influence of thickness of the sample fabrics on their bending rigidity.

Table 29. ANOVA for fabric thickness and bending rigidity

	df	SS	MS	F	P-value
Regression	1	115.5116	115.5116	52592.72	2.17E-09
Residual	4	0.008785	0.002196		
Total	5	115.5204			

Table 30. Correlation between fabric thickness and bending rigidity

Correlation equation	BR = 31.627FT - 0.5761 BR = Bending rigidity FT = fabric thickness
Pearson correlation coefficient (r)	+ 0.9999
Coefficient of determination (R^2)	0.9999

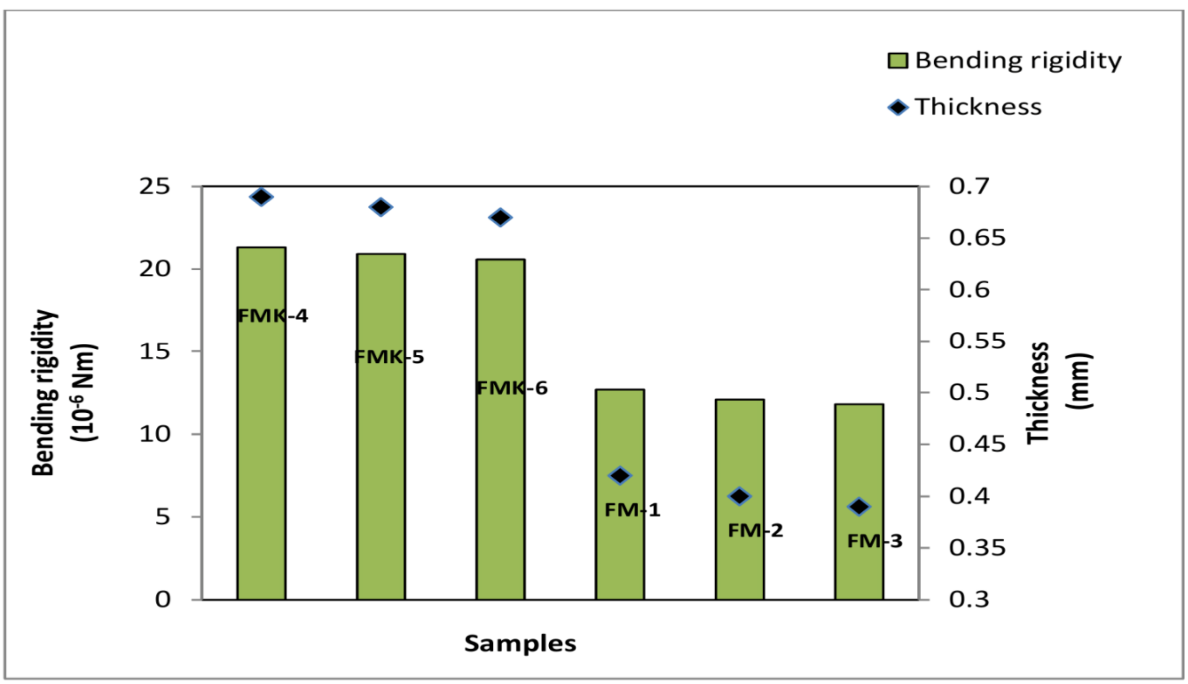

Figure 35. Fabric thickness vs. bending rigidity.

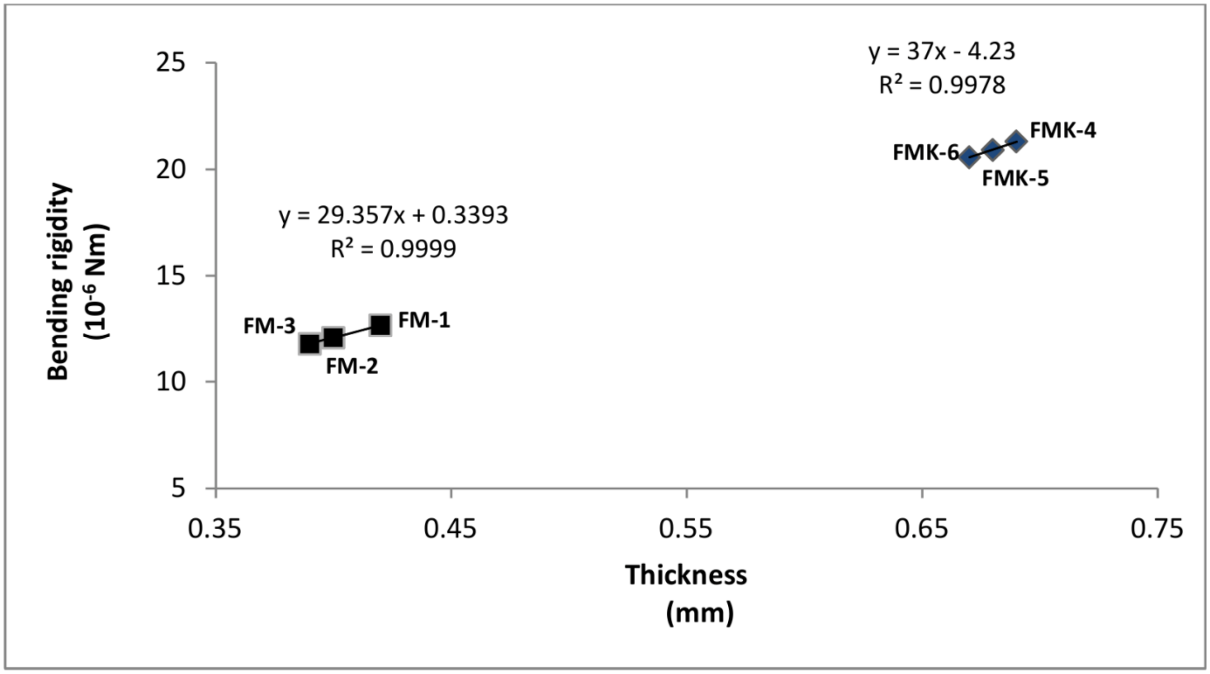

Figure 36. Effect of fabric thickness on bending rigidity for two-layered and three-layered samples.

However, bending rigidity values of two-layered samples are lower than bending rigidity values of three-layered samples due to their lower thickness values than three-layered samples. Among all the samples, highest bending rigidity is obtained in case of sample FMK-4 due to its highest fabric thickness property. And lowest bending rigidity is found for the sample fabric FM-3 due to its lowest thickness property. When only two-layered three samples are compared to each other, higher bending rigidity is obtained for FM-1 sample due to its higher thickness value than FM-2 and FM-3. Again, when only three-layered three samples are compared to each other, lower bending rigidity is found in case of sample FMK-6 due to its lower thickness value than the rest two other samples FMK-4 and FMK-5.

4.1.7 Analysis of breaking strength

Breaking force was measured to evaluate the breaking strength property of the sample fabrics. From ANOVA of Table-31, P-value is found less than 0.05 which explains a significant influence of fabric weight on the breaking force of the samples. Pearson correlation coefficient (r) and Coefficient of determination (R^2) for fabric weight and breaking force are obtained

+0.9523 and 0.9068 respectively from Table-32. These results represent a strong positive correlation between fabric weight and breaking force of the laminated sample fabrics. Again, P-value is obtained less than 0.05 from ANOVA of Table-33 for fabric density and breaking force that indicates a significant influence of fabric density on breaking force of the samples. From Table-34, r-value and R^2-value are obtained +0.9984 and 0.9969 respectively. This also explains a strong positive linear relationship between fabric density and breaking force. So, it can be said from this statistical analysis that breaking strength of the laminated fabric increases with the increase of fabric weight and density.

However, from the test results (Table-5) and Figures (37, 38, 39 & 40), it is clear that breaking forces for all three-layered samples are more than breaking forces of all two-layered samples. This is because of increasing fabric weight and fabric density of three-layered three samples after adding polyester knitted fabrics as their inner layers during lamination process. Among the all six samples, highest breaking force value is obtained for FMK-4 sample due to its highest fabric weight and density and lowest is obtained for FM-3 sample due to its lowest fabric weight and density. When only two-layered three samples are compared to each other, the best breaking force is obtained for FM-1 sample and lowest is obtained for FM-3 sample. Here the reason is not only the fabric weight and fabric density, but also the warp cover factor and weft cover

Table 31. ANOVA for fabric weight and breaking force

	df	**SS**	**MS**	**F**	**P-value**
Regression	1	8083.028	8083.028	38.94001	0.003361
Residual	4	830.3056	207.5764		
Total	5	8913.333			

Table 32. Correlation between fabric weight and breaking force

Correlation equation	BF = 0.5891FW + 267.33 BF = Breaking force FW = Fabric weight
Pearson correlation coefficient (r)	+ 0.9523
Coefficient of determination (R^2)	0.9068

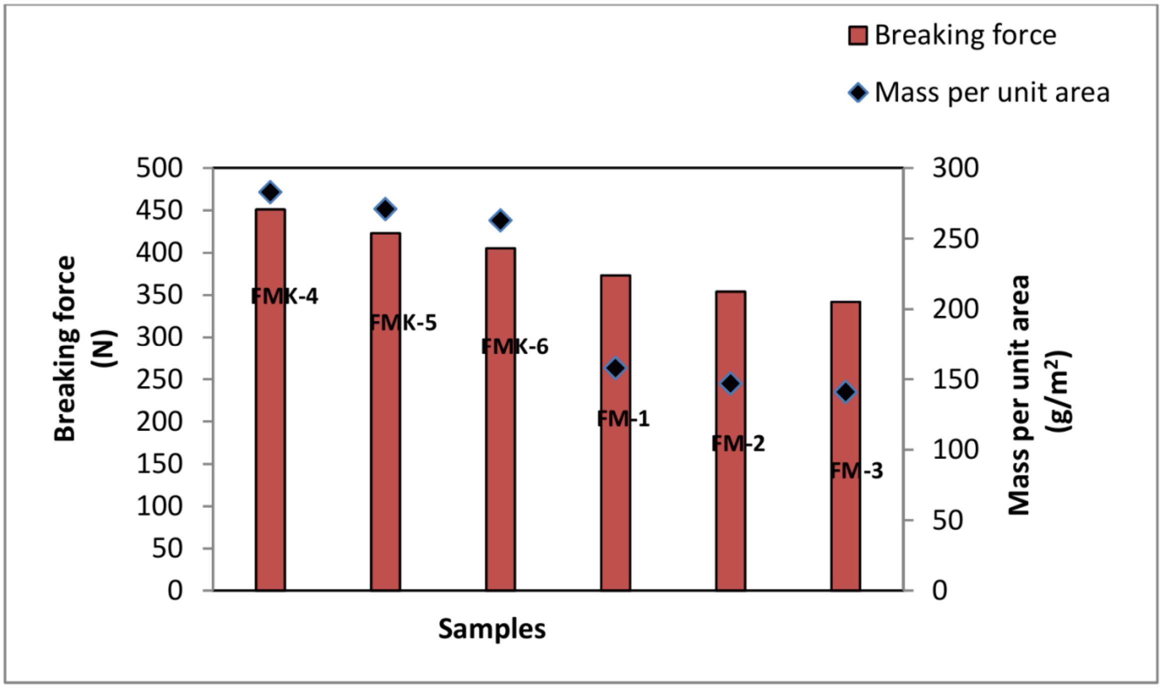

Figure 37. Fabric weight vs. breaking force.

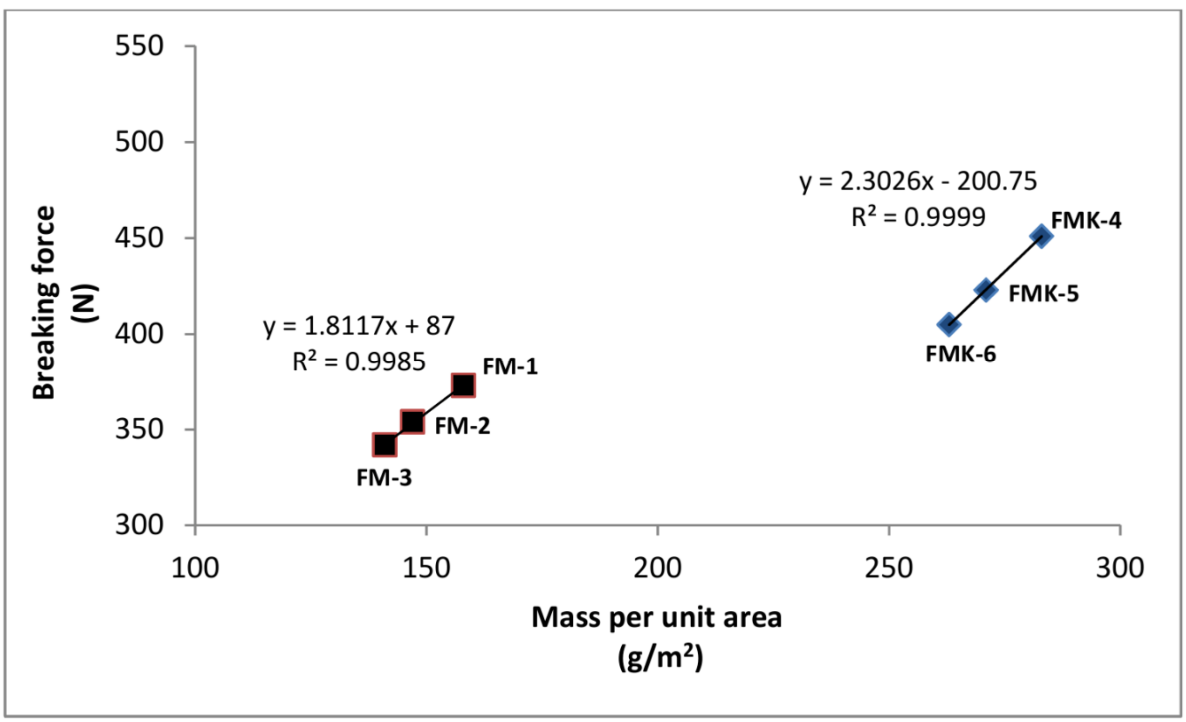

Figure 38. Effect of fabric weight on breaking force for two-layered and three-layered samples.

Table 33. ANOVA for fabric density and breaking force

	df	SS	MS	F	P-value
Regression	1	8885.367	8885.367	1270.872	3.7E-06
Residual	4	27.96621	6.991553		
Total	5	8913.333			

Table 34. Correlation between fabric density and breaking force

Correlation equation	BF = 2.219FD - 461.66 BF = Breaking force FD = fabric density
Pearson correlation coefficient (r)	+ 0.9984
Coefficient of determination (R^2)	0.9969

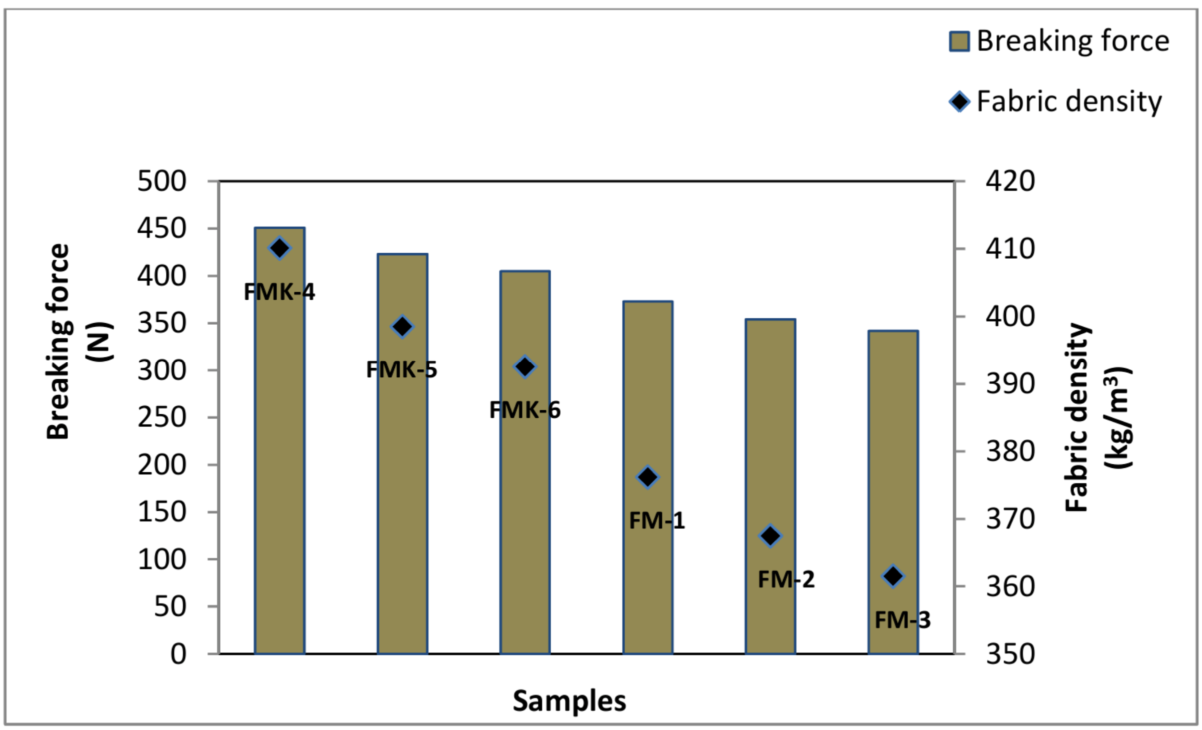

Figure 39. Fabric density vs. breaking force.

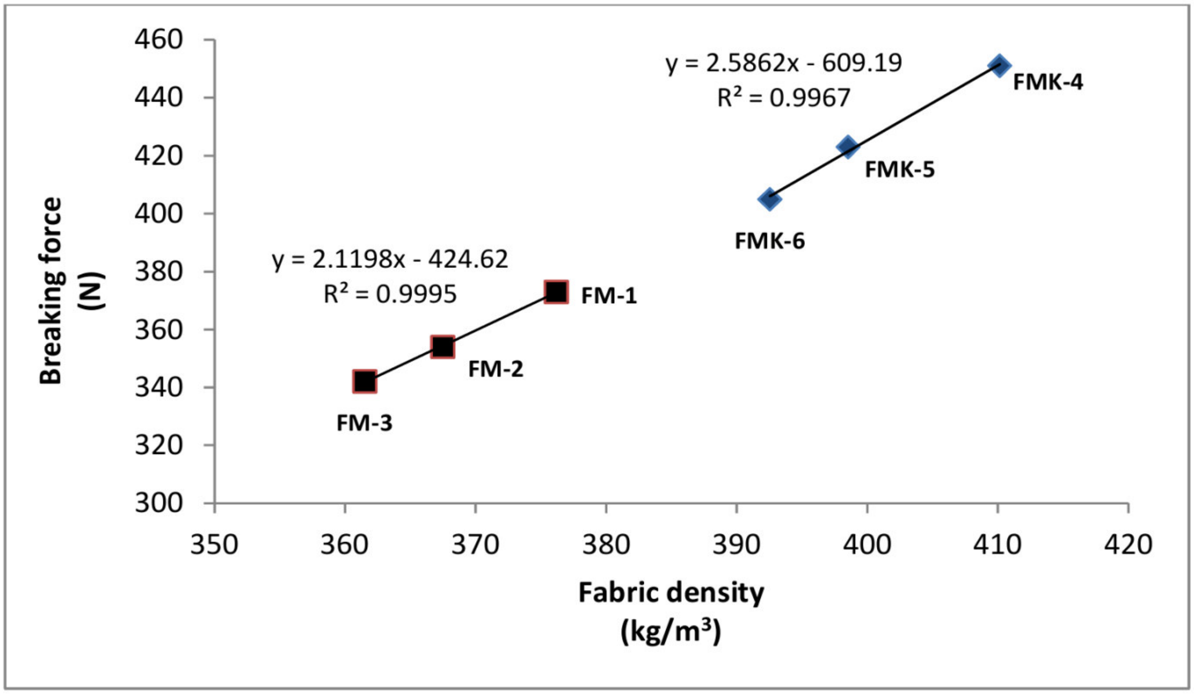

Figure 40. Effect of fabric density on breaking force for two-layered and three-layered samples.

factor of outer layer woven part of FM-1 sample that are higher than the cover factors of outer layer parts of FM-2 and FM-3 samples. Again, when only three-layered three samples are compared to each other, the best breaking force is obtained for FMK-4 sample and the lowest is obtained for FMK-6 sample. The reason is also here not only the fabric weight and density, but also warp cover factor and weft cover factor of outer layer woven part of FMK-4 sample that are higher than the cover factors of outer layer woven parts of FMK-5 and FMK-6 samples.

4.2 Results and discussion for evaluation of enhanced hydrostatic resistance and mechanical performance of PTFE membrane laminated fabrics

4.2.1 Analysis of test results for WMK-2 sample fabric after coating

4.2.1.1 Analysis of coating for WMK-2 sample fabric

Table-7 shows the different characteristics of WMK-2 sample fabric which was coated under different mixing ratios of C_6-based fluorocarbon chemical (®RUCOSTAR EEE6) and polysiloxane hydrophobic softening agent (®RUCOFIN HSF) according to T-2, T-3, T-4, T-5, T-6 and T-7. Here in the table, the highest increase in percentage for areal density (g/m^2) after coating was obtained 10.78% for T-3 sample. Consequently highest fabric density (Kg/m^3) was also obtained for T-3 sample after coating when 50 g/L ®RUCOSTAR EEE6 was added with 15 g/L ®RUCOFIN HSF in the coating solution. However, when 60 g/L ®RUCOSTAR EEE6 was added with 15 g/L ®RUCOFIN HSF (T-4 sample) and 60 g/L ®RUCOSTAR EEE6 was added with 20 g/L ®RUCOFIN HSF (T-7 sample), then increases in percentage for areal density after coating were 5.99% and 5.39% respectively. Because when fluorocarbon chemical concentration here increased, then liquor penetration percentage decreased.

4.2.1.2 Water-repellent property for coated WMK-2 sample fabric

Water repellent property is one of the important properties for outdoor sports fabrics. More water- repellent property of a fabric shows more resistant property against the wetting by water. In spray test results, there are six ratings shown in a photographic chart according to AATCC 22 method [68]. A specimen with complete wetting of the entire face is assigned by "0" rating, while a specimen with no sticking or wetting of the face is assigned by "100" rating. The rest four ratings are in between "0" to "100" ratings. After coating of WMK-2 sample with different mixing ratios, spray test ratings are obtained like the following:

Table 35. Spray test ratings of coated WMK-2 sample with different mixing ratios

Sample fabric	Spray rating
T-2 (40 g/L ®RUCOSTAR EEE6 with 15 g/L ®RUCOFIN HSF)	100
T-3 (50 g/L ®RUCOSTAR EEE6 with 15 g/L ®RUCOFIN HSF)	100
T-4 (60 g/L ®RUCOSTAR EEE6 with 15 g/L ®RUCOFIN HSF)	90
T-5 (40 g/L ®RUCOSTAR EEE6 with 20 g/L ®RUCOFIN HSF)	100
T-6 (50 g/L ®RUCOSTAR EEE6 with 20 g/L ®RUCOFIN HSF)	100
T-7 (60 g/L ®RUCOSTAR EEE6 with 20 g/L ®RUCOFIN HSF)	90

4.2.1.3 Analysis of other properties for WMK-2 sample fabric after coating with different mixing ratios

Highest increase of hydrostatic resistance is obtained in case of sample T-3 from Figure-41 and highest breaking force is also found for T-3 sample from Figure-42, when the test results of WMK-2 coated samples with different mixing ratios are compared. The reason is the highest fabric density of T-3 sample after coating that is obtained from Table-7. After coating, bending rigidity is also found higher for T-3 sample from Figure-43, but not so big differences in values are found. On the other hand, after coating the evaporative resistance values range from 8.52 to 8.84 m^2Pa/W from Figure-44 and this indicates no big differences in evaporative resistance values after coating. And from Figure-45, no big differences are obtained in air permeability values for WMK-2 samples after coating with different mixing ratios.

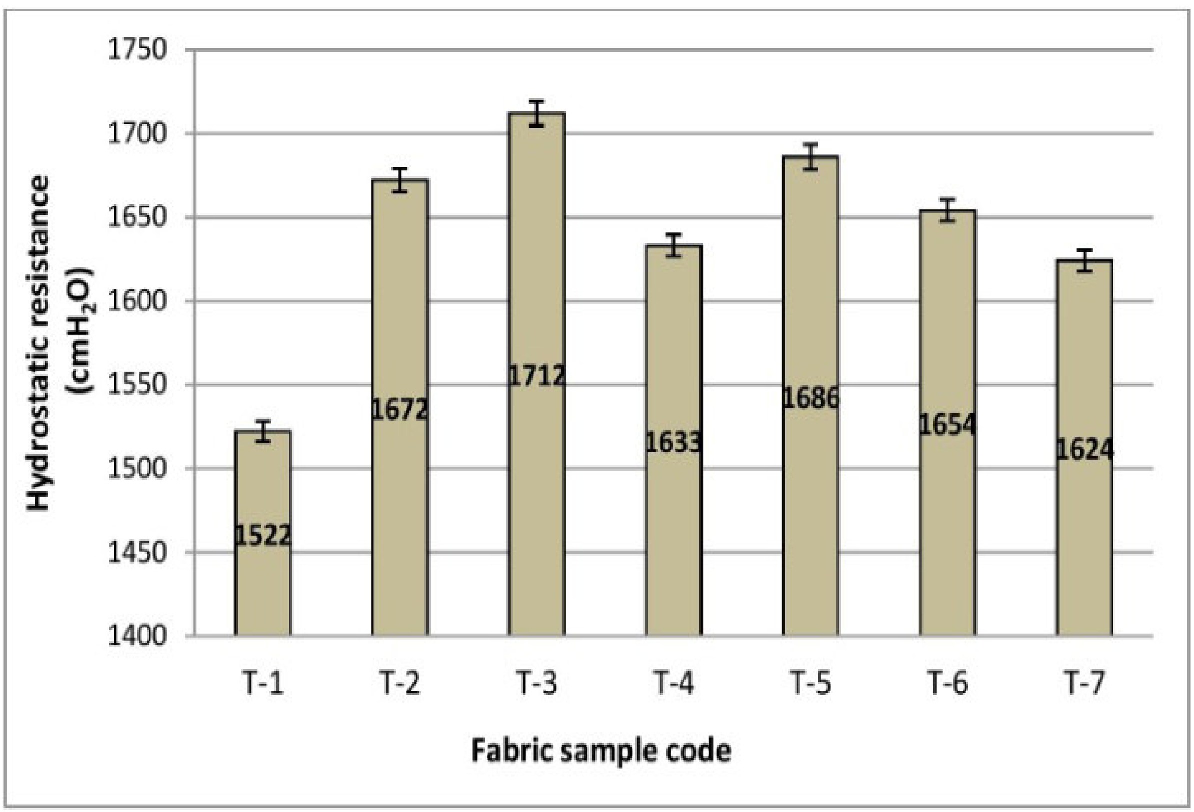

Figure 41. Hydrostatic resistance after coating with different ratios for WMK-2 sample.

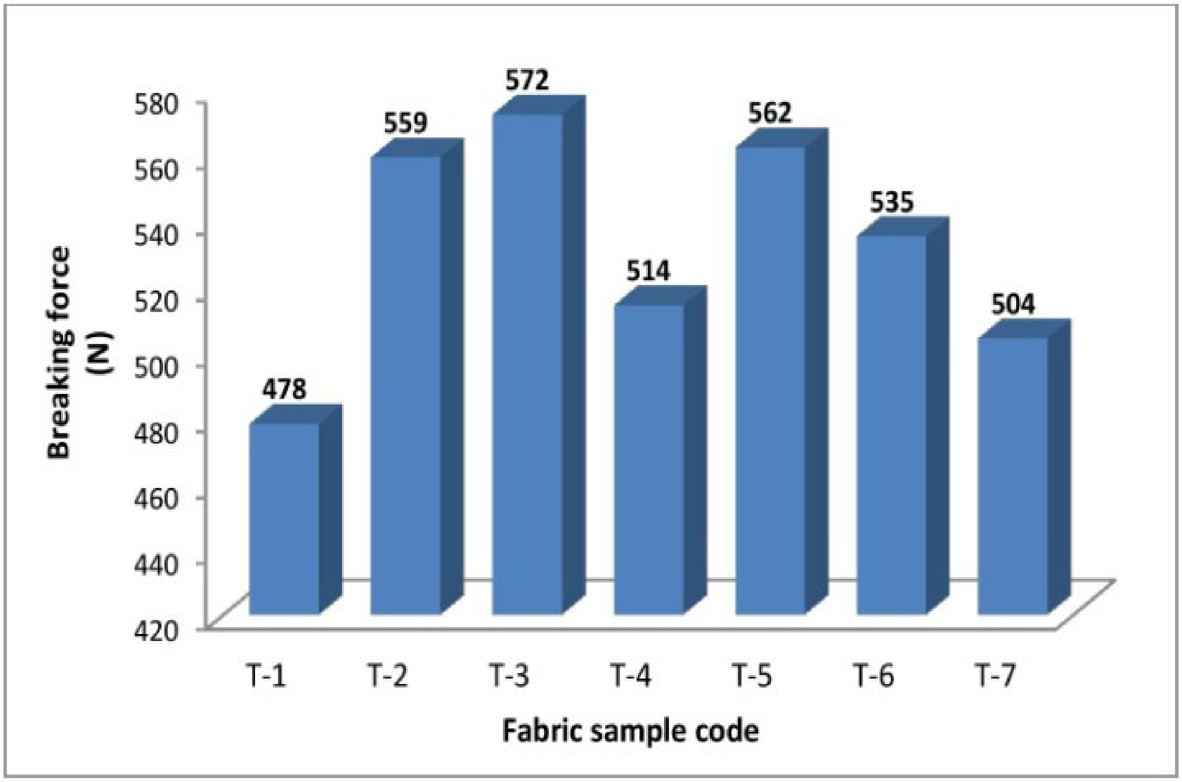

Figure 42. Breaking force after coating with different ratios for WMK-2 sample.

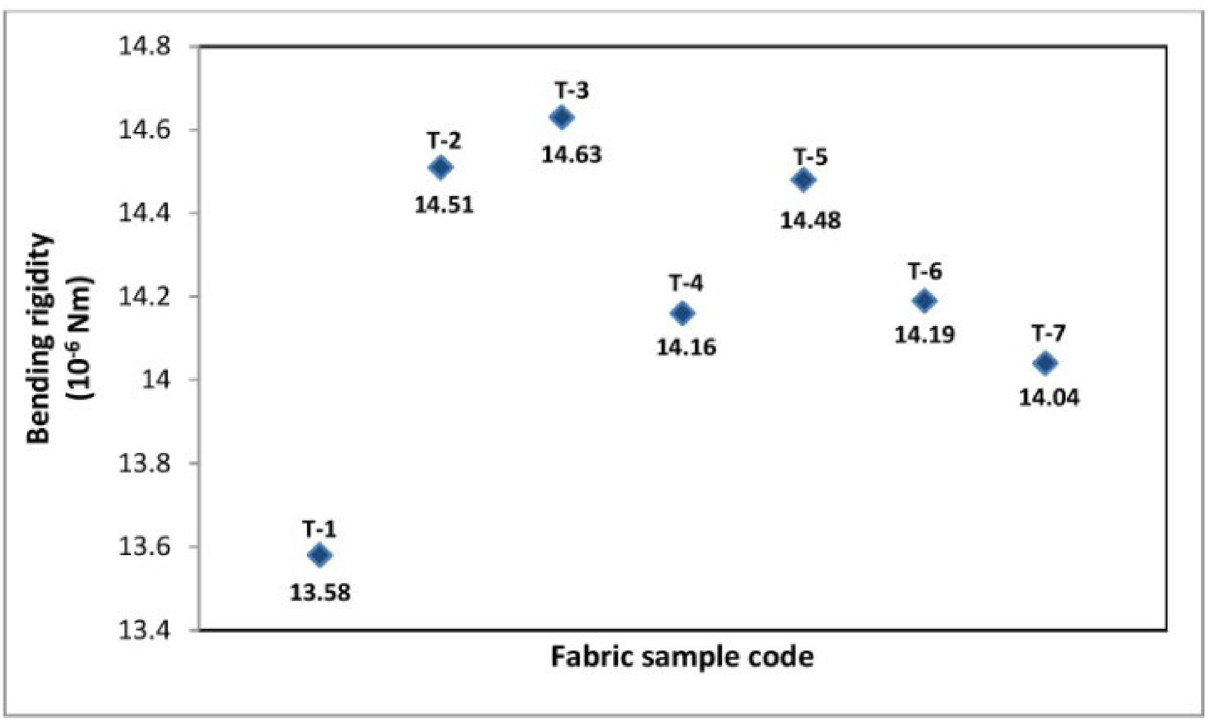

Figure 43. Bending rigidity after coating with different ratios for WMK-2 sample.

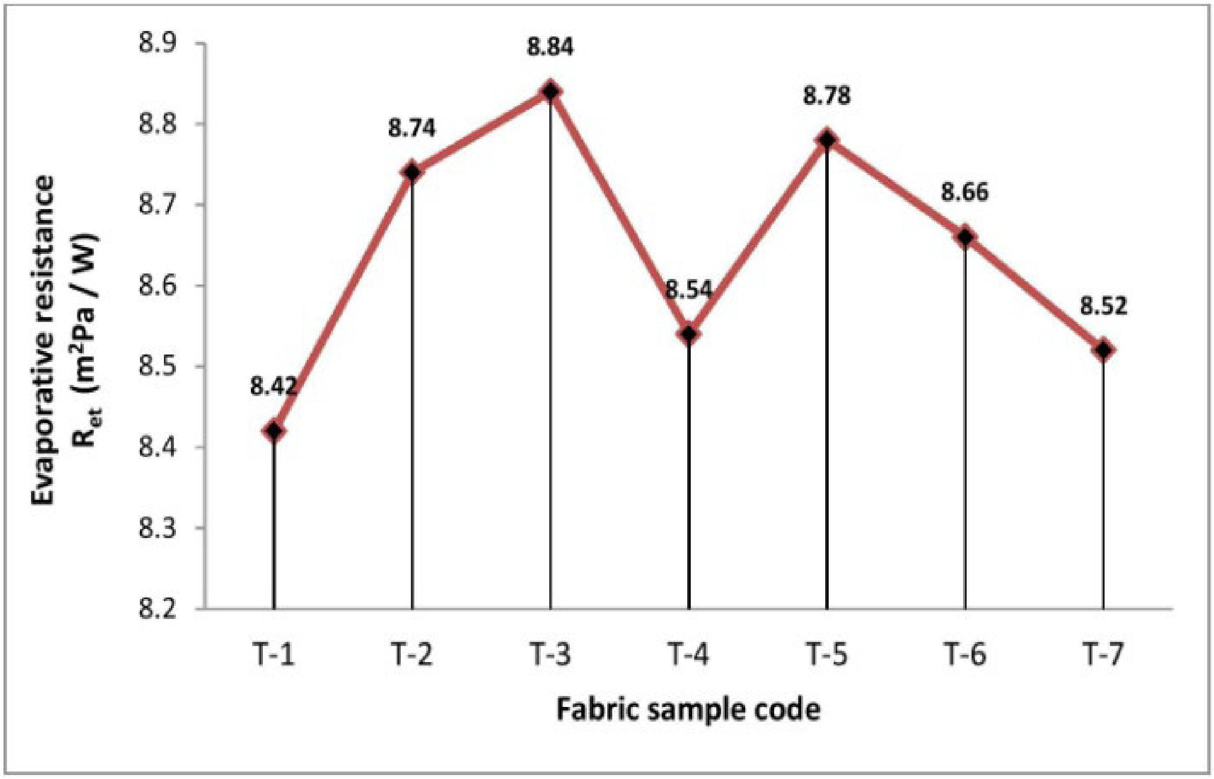

Figure 44. Evaporative resistance after coating with different ratios for WMK-2 sample.

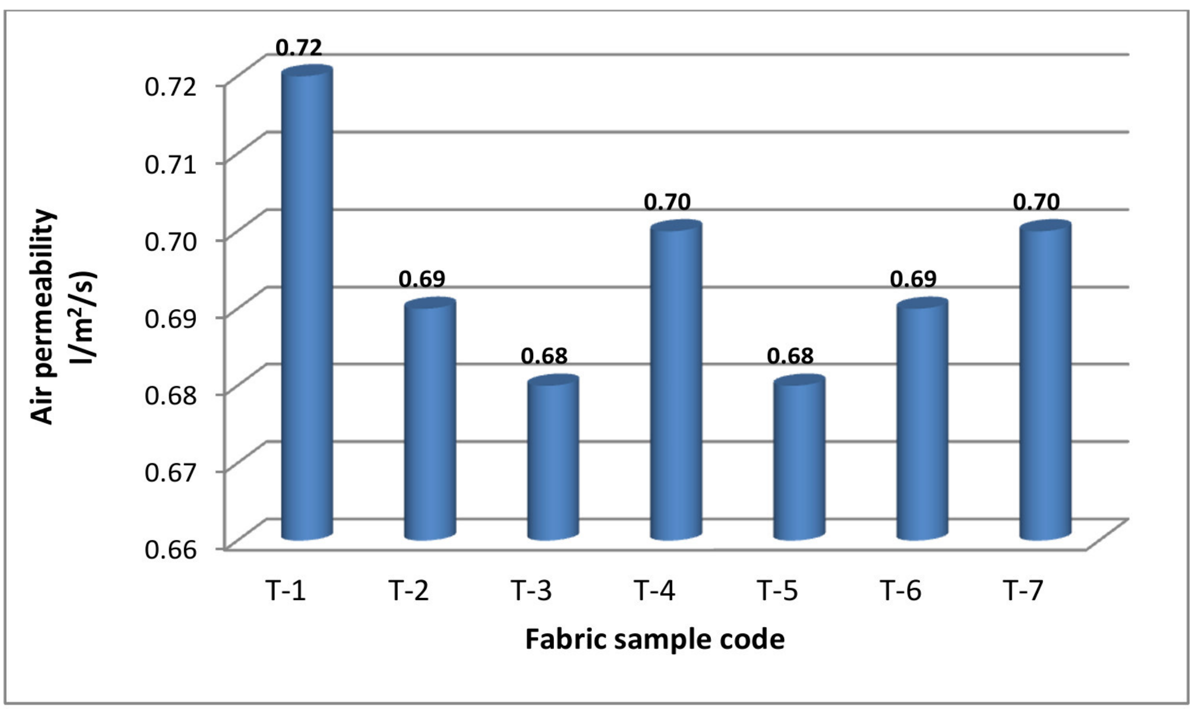

Figure 45. Air permeability after coating with different ratios for WMK-2 sample.

After analysis of the all above test results for coated WMK-2 sample fabric with different mixing ratios, it has been found that the mixing ratio of T-3 sample gives the best results. As a result, this ratio, i.e., 50 g/L ®RUCOSTAR EEE6 with 15 g/L ®RUCOFIN HSF was selected as a standard recipe for coating other three-layered PTFE membrane laminated waterproof breathable fabrics, like, WMK-1, WMF-3 and WMF-4 samples.

4.2.2 Evaluation of test results for PTFE membrane laminated four different types of samples after coating

4.2.2.1 Analysis of applied coating

Coating solution that was applied on four different types of PTFE membrane laminated fabrics, was prepared with 50 g/L C_6-based fluorocarbon water-repellent chemical (®RUCOSTAR EEE6) and 15 g/L polysiloxane hydrophobic softening agent (®RUCOFIN HSF). Among the four samples, WMK-1 and WMK-2 are more densely fabrics. WMK-2 is more densely than WMK-1 due to its more densely outer woven layer and inner knitted layer. WMF-3 and WMF-4 are less densely due to their higher thickness and inner fleece knitted structures. Higher increases in areal density (g/m^2) after coating are obtained in case of WMK-1 and WMK-2 which are 11.24% and 10.78% increases respectively (Table-9). On the other hand, 7.96% and 7.46% increases are found for the samples of WMF-3 and WMF-4. The reason for less increase in this case may be due to their inner fleece knitted structures which cannot hold the coating solution as properly as the first two samples. However, fabric thickness values of all four fabrics also increase after coating and these values influence on their total fabric density values as well.

4.2.2.2 Morphology and water-repellent property

Scanning electron microscope (SEM) gives a proper idea about morphological cross-sectional images of four different laminated fabrics before and after coating. It is evident from the images that WMK-1, WMK-2, WMF-3 and WMF-4 have three layers and their membranes are between outer and inner layers (Figure-46). However, the coated samples show more regular and smooth surfaces under scanning electron microscope. This is due to the evenly deposition of coating materials on the fabric layers, as a result, individual yarns within the fabrics are more strongly attached to each other which can contribute to the higher resistance against water pressure as well as can increase their breaking strength.

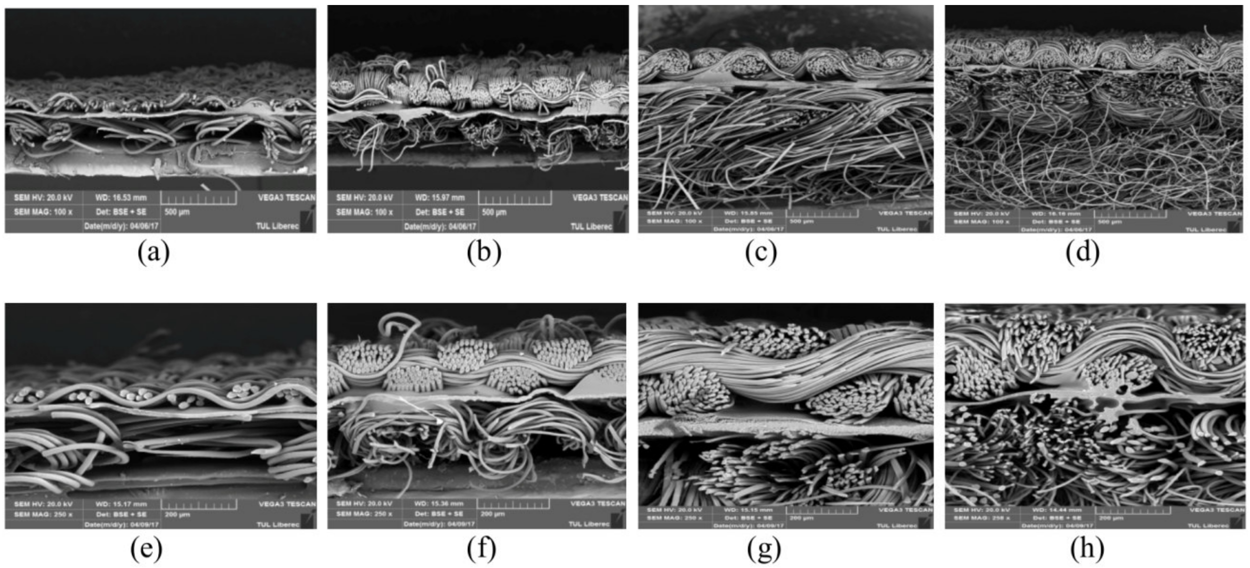

Figure 46. SEM images of uncoated samples: (a) WMK-1 (b) WMK-2 (c) WMF-3 (d) WMF-4 and coated samples: (e) WMK-1 (f) WMK-2 (g) WMF-3 (h) WMF-4.

Water-repellent property of four different types of laminated sample fabrics was measured by the evaluation of spray test results according to AATCC 22 method [68]. At first, all four coated samples showed “100” rating. Then all four coated samples were washed with 4 g/l detergent at 40°C for 1 hour. After five washes, water-repellent property was again evaluated by spray test results. Here also all the four samples showed “100” rating because of complete non-wetting property of the coated fabrics.

4.2.2.3 Comparison of hydrostatic resistance

From Figure-47, it is evident that there is a good positive relation between fabric density and hydrostatic resistance. Hydrostatic resistance increases with the increase of fabric density in case of both coated and uncoated fabrics. Highest hydrostatic resistance is obtained for WMK-2 sample in both cases (Table-10). This is due to its higher fabric density and more compact structure than any other sample. For example, warp cover factor and weft cover factor of its outer woven part and stitch density of its inner knitted part are denser than any other PTFE membrane laminated fabric sample. Hydrostatic resistance values of WMF-3 and WMF-4 are less than the hydrostatic resistance values of others. The reason is their lower fabric density. Moreover, their inner fleece knitted parts cannot make higher resistance against water pressure, though their thickness values are higher than others. So, hydrostatic resistance is attributed here to fabric density as well as compactness of fabric structure. However, the comparisons between coated and uncoated samples from ANOVA of Table-36 express that there are significant increases of hydrostatic resistance after coating for all samples. Because, P-values for all samples are less than 0.05 and F-ratios are clearly higher than F-critical values. Increases of hydrostatic resistance after coating for WMK-1 and WMK-2 samples are 152 cmH_2O and 190 cmH_2O respectively which are about 10.38% and 12.48% increases from uncoated samples. About 9.66% increase for WMF-3 and 9.16% increase for WMF-4 are found from hydrostatic resistance test results of Table-10. These are also significant increases after coating. So, it can be said that there are significant enhancement of hydrostatic resistance for all laminated sample fabrics after coating.

Table 36. ANOVA for hydrostatic resistance of coated and uncoated sample fabrics

Compared pairs (coated and uncoated)	Source of Variation	SS	df	MS	F-ratio	P-value	F -critical
WMK-1	Between Groups	34504.17	1	34504.17	444.26	2.995E-05	7.709
	Within Groups	310.66	4	77.66			
	Total	34814.83	5				
WMK-2	Between Groups	54150	1	54150	786.68	9.613E-06	7.709
	Within Groups	275.33	4	68.83			
	Total	54425.33	5				
WMF-3	Between Groups	16537.50	1	16537.50	351.86	4.756E-05	7.709
	Within Groups	188	4	47			
	Total	16725.50	5				
WMF-4	Between Groups	15606	1	15606	373.05	4.235E-05	7.709
	Within Groups	167.33	4	41.83			
	Total	15773.33	5				

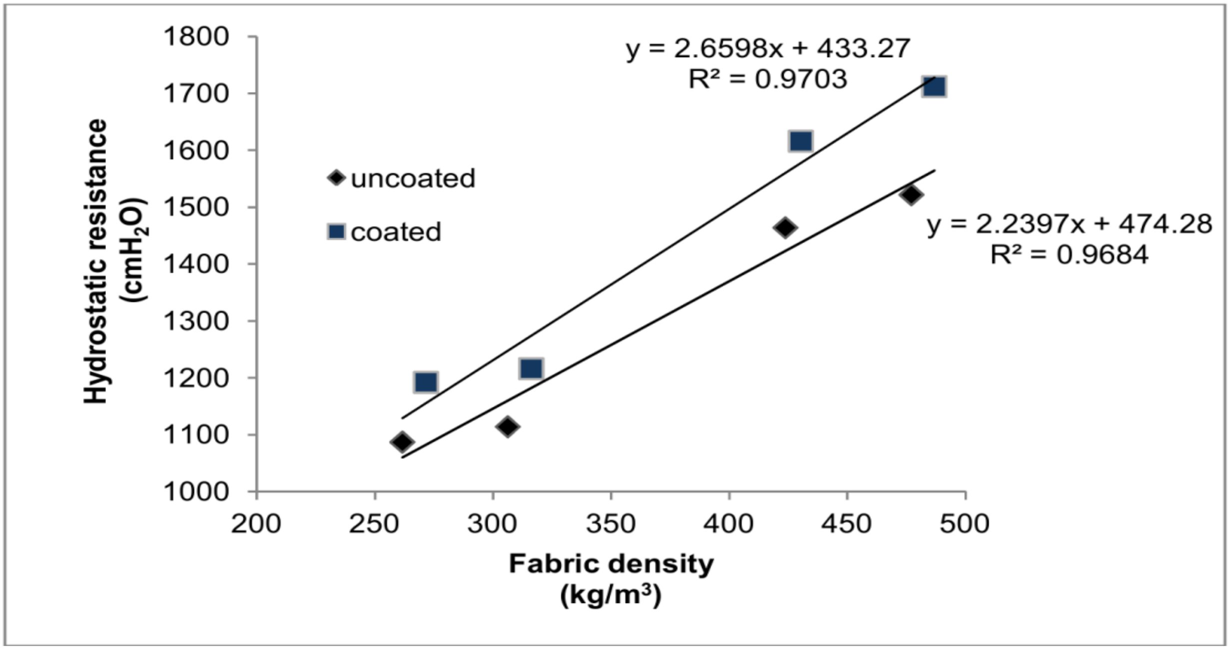

Figure 47. Hydrostatic resistance of coated and uncoated samples.

4.2.2.4 Comparison of breaking strength

Breaking strength test was performed to evaluate the mechanical property of the sample fabrics. Breaking strength was determined by breaking force. From the test results of Table-10, ANOVA of Table-37 and Figure-48, it is evident that there are significant increases of breaking strength of all samples after coating. Here, ANOVA table shows that F-ratios are higher than F-critical values and P-values are less than 0.05 in all fabrics when there are comparisons between coated and uncoated samples. The highest increase in breaking strength after coating among all samples is found for WMK-2 sample. Here, about 19.67% increase is obtained after coating because of its denser and more compact structure than others. About 16.34%, 15.73% and 14.78% increases are found in case of WMK-1, WMF-3 and WMF-4 samples respectively. From Figure-48, it is also clear that there is a positive relationship between fabric density and breaking strength. Breaking strength increases with the increase of fabric density and this trend is observed for both coated and uncoated samples. WMK-2 and WMK-1 samples show higher breaking strength property than WMF-3 and WMF-4 samples due to their higher fabric density values in both cases.

Table 37. ANOVA for breaking strength of coated and uncoated sample fabrics

Compared pairs (coated and uncoated)	Source of Variation	SS	df	MS	F-ratio	P-value	F -critical
WMK-1	Between Groups	11424.40	1	11424.40	179.21	9.27894E-07	5.318
	Within Groups	510	8	63.75			
	Total	11934.40	9				
WMK-2	Between Groups	21902.40	1	21902.40	271.57	1.85467E-07	5.318
	Within Groups	645.20	8	80.65			
	Total	22547.60	9				
WMF-3	Between Groups	8065.60	1	8065.60	83.32	1.67004E-05	5.318
	Within Groups	774.40	8	96.80			
	Total	8840	9				
WMF-4	Between Groups	8065.60	1	8065.60	65.55	4.00664E-05	5.318
	Within Groups	984.40	8	123.05			
	Total	9050	9				

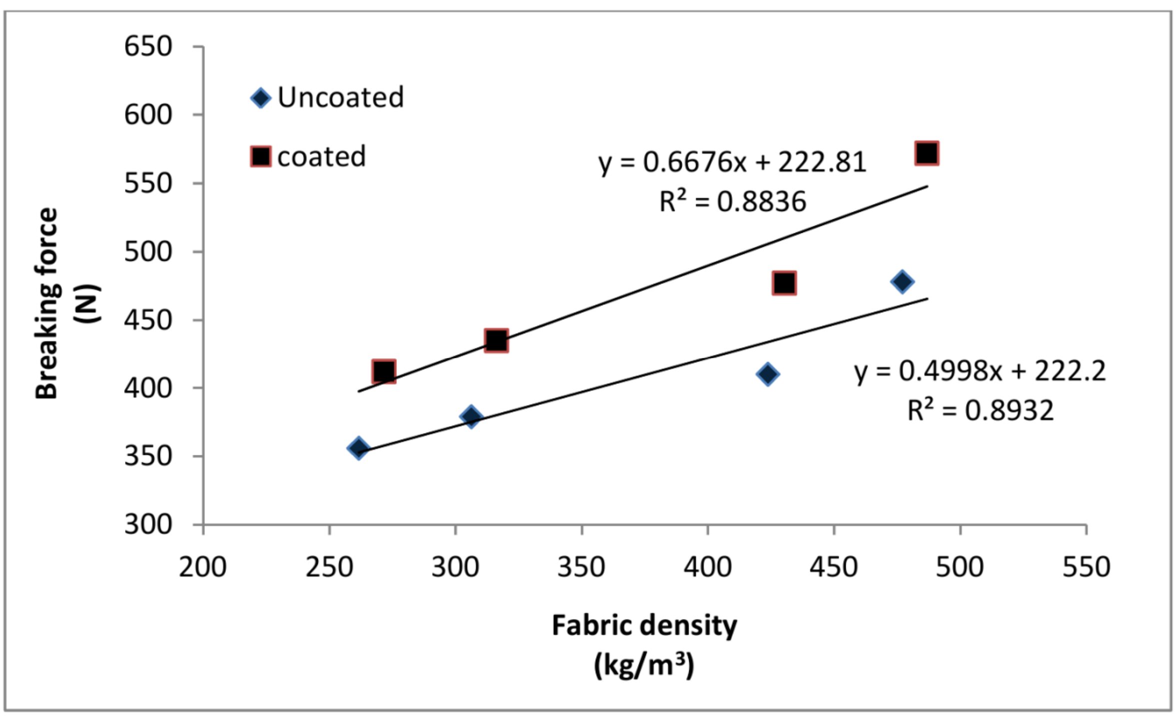

Figure 48. Breaking force of coated and uncoated samples.

4.2.2.5 Comparison of bending rigidity

Stiffness is a special property of a fabric to keep it standing without support. It is determined by bending rigidity which is an important comfort parameter. A fabric needs stiffness, but very stiff fabric can be uncomfortable and unfit for use. When coated and uncoated samples are compared, ANOVA from Table-38 shows that F-ratios are higher than F-critical values in case of all samples as well as P-values are less than 0.05. But, from bending rigidity test results of Table-10 and Figure-49, it can be said that increases of bending rigidity after coating are not as high as the increases of hydrostatic resistance and tensile strength after coating. Here, maximum increase is obtained 7.73% for WMK-2 sample after coating. 5.95%, 5.26% and 5.09% increases are found for WMK-1, WMF-3 and WMF-4 samples respectively. These results reveal that there are not much higher increases in stiffness property after coating because of using polysiloxane hydrophobic softening agent in the coating solution.

Table 38. ANOVA for bending rigidity of coated and uncoated sample fabrics

Compared pairs (coated and uncoated)	Source of Variation	SS	df	MS	F-ratio	P-value	F -critical
WMK-1	Between Groups	0.497	1	0.497	12.689	0.0073771	5.318
	Within Groups	0.314	8	0.039			
	Total	0.811	9				
WMK-2	Between Groups	2.809	1	2.809	138.682	2.474E-06	5.318
	Within Groups	0.162	8	0.020			
	Total	2.971	9				
WMF-3	Between Groups	5.169	1	5.169	175.063	1.015E-06	5.318
	Within Groups	0.236	8	0.029			
	Total	5.405	9				
WMF-4	Between Groups	5.127	1	5.127	292.112	1.396E-07	5.318
	Within Groups	0.140	8	0.018			
	Total	5.267	9				

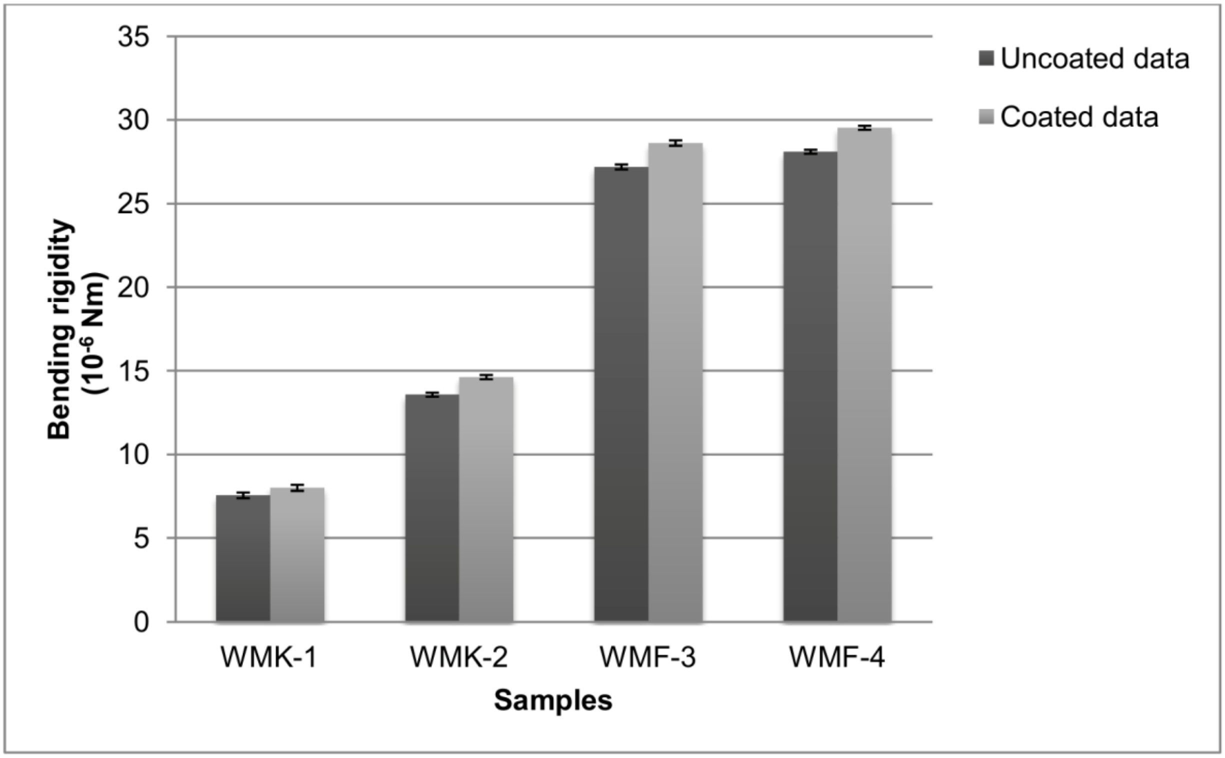

Figure 49. Bending rigidity of coated and uncoated samples.

4.2.2.6 Comparison of water vapour permeability

By measuring evaporative resistance (R_{et}), water vapour permeability of a fabric is obtained. Higher R_{et} value determines the lower water vapour transmission of the measuring sample. The effect of fabric weight on water vapour permeability is shown in Figure-50 for both coated and uncoated samples. It is clear that R_{et} values of all samples increase with the increases of fabric weights in both cases. The fabric becomes uncomfortable with increasing the fabric weight and evaporative resistance. Among the four samples, WMF-3 and WMF-4 samples have the lower water vapour permeability due to their higher R_{et} values. The reason is their higher fabric weights and inner knitted fleece structures which cause more air entrapment preventing the diffusion rate of water vapour. As a result, it can be difficult to release sweat from the body in the form of water vapour. On the other hand, WMK-1 and WMK-2 samples show better water vapour permeability due to their lower fabric weights. Again, WMK-1 sample has less densely outer woven structure and less stitch density of inner knitted structure along with lower thickness and lower fabric weight which make it best water vapour permeable among all the samples. The more water vapour transmission of this fabric results in higher fabric breathability. From ANOVA of Table-39, it can be said that there are no significant changes in R_{et} values for all four samples after coating because F-ratios are lower than F-critical values and P-values are more than 0.05. The reason is that applied finish, like, hydrophobic water-repellent coating to a fabric has no great effect on the diffusion process [71]. As a result, no significant change in water vapour permeability is obtained after coating for any sample.

Table 39. ANOVA for evaporative resistance (R_{et}) of coated and uncoated sample fabrics

Compared pairs (coated and uncoated)	Source of Variation	SS	df	MS	F-ratio	P-value	F -critical
WMK-1	Between Groups	0.225	1	0.225	2.885	0.128	5.318
	Within Groups	0.624	8	0.078			
	Total	0.849	9				
WMK-2	Between Groups	0.441	1	0.441	4.20	0.075	5.318
	Within Groups	0.84	8	0.105			
	Total	1.281	9				
WMF-3	Between Groups	0.196	1	0.196	2.904	0.127	5.318
	Within Groups	0.54	8	0.068			
	Total	0.736	9				
WMF-4	Between Groups	0.144	1	0.144	1.882	0.207	5.318
	Within Groups	0.612	8	0.077			
	Total	0.756	9				

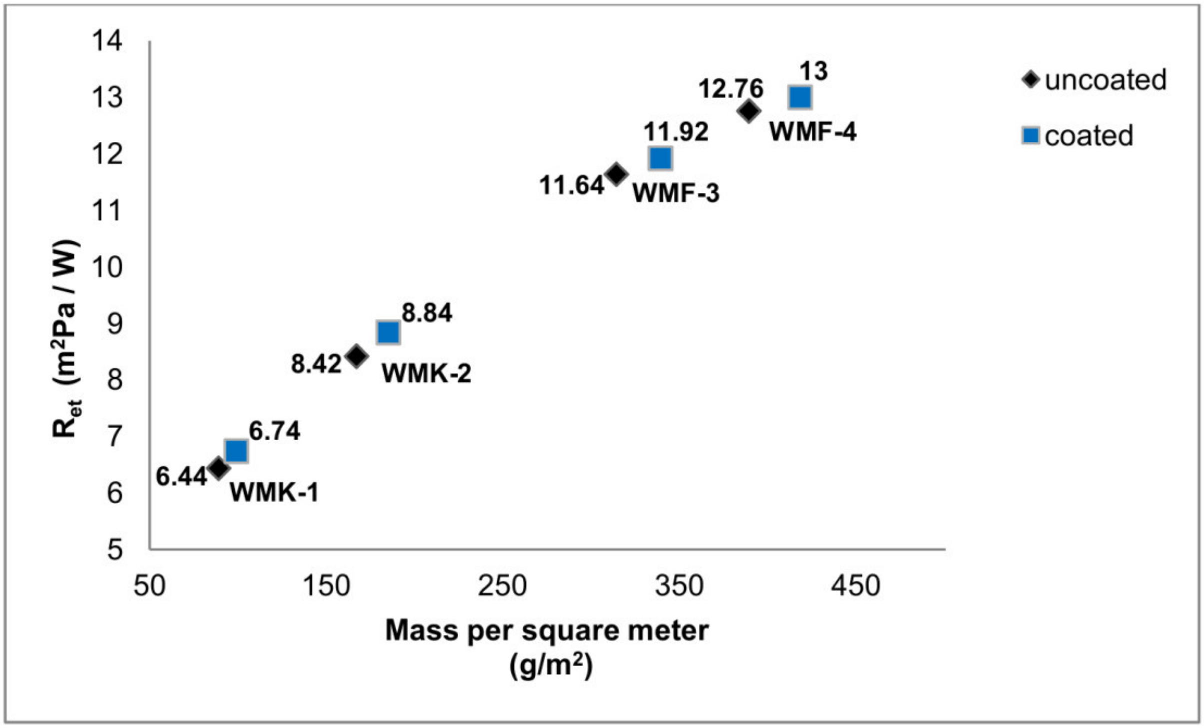

Figure 50. Evaporative resistance (R_{et}) of coated and uncoated samples.

4.2.2.7 Comparison of air permeability

In order to determine the changes of air permeability after coating for all samples, analysis of variance of Table-40 and Figure-51 can be discussed. From ANOVA table, F-ratios of all samples are lower than F-critical values and P-values are more than 0.05 which indicates that there are no significant changes in air permeability after coating for all fabric samples. Figure-51 also reveals the same appearance that there are actually on big differences in air permeability between coated and uncoated samples. So, it can be said that the effects of applied water-repellent coating solution on air permeability of different PTFE laminated fabrics are not significant.

Table 40. ANOVA for air permeability of coated and uncoated sample fabrics

Compared pairs (coated and uncoated)	Source of Variation	SS	df	MS	F-ratio	P-value	F -critical
WMK-1	Between Groups	0.005	1	0.005	2.454	0.135	4.414
	Within Groups	0.035	18	0.002			
	Total	0.040	19				
WMK-2	Between Groups	0.008	1	0.008	3.113	0.095	4.414
	Within Groups	0.044	18	0.002			
	Total	0.052	19				
WMF-3	Between Groups	0.019	1	0.019	4.366	0.051	4.414
	Within Groups	0.082	18	0.005			
	Total	0.102	19				
WMF-4	Between Groups	0.019	1	0.019	3.793	0.067	4.414
	Within Groups	0.091	18	0.005			
	Total	0.110	19				

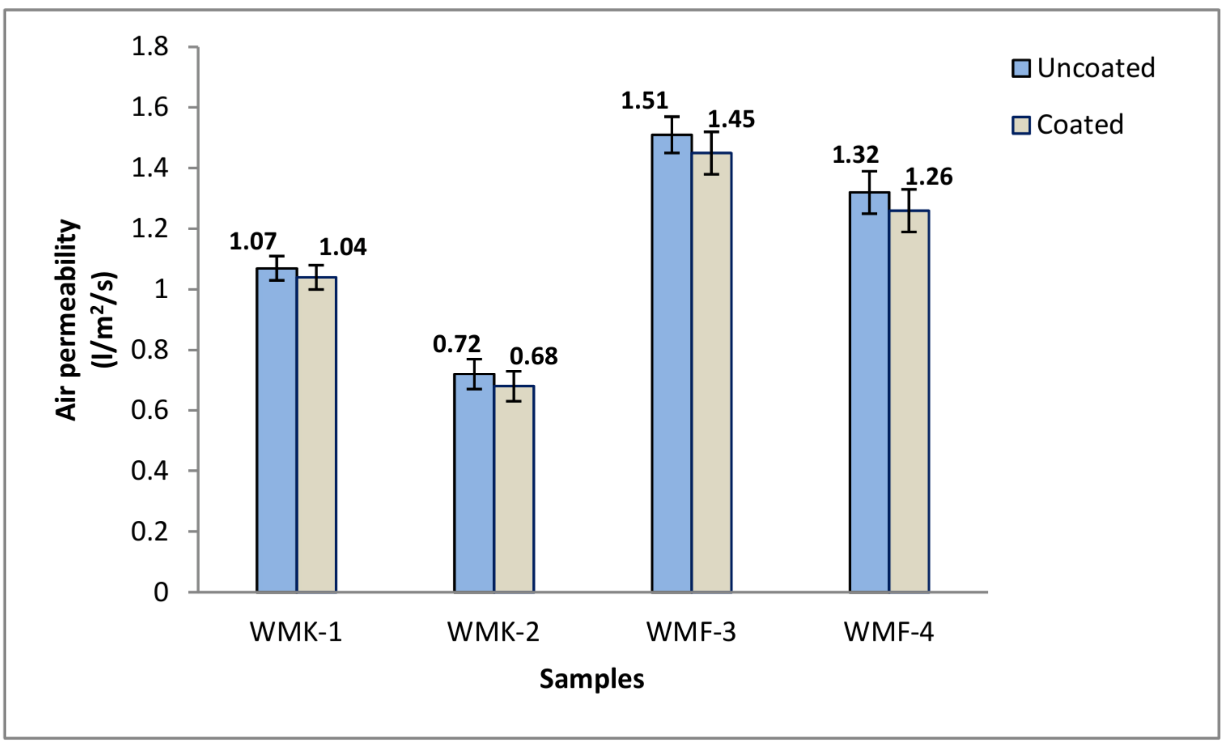

Figure 51. Air permeability of coated and uncoated samples.

CHAPTER 5: SUMMARY AND CONCLUSIONS

This chapter summarizes the research work that was carried out in the thesis. This study newly designed and prepared microporous PU membrane laminated waterproof breathable fabrics with different layered structures that could be used as outdoor sports fabrics and their different properties, like, hydrostatic resistance, thermo-physiological comfort properties and mechanical behaviors were statistically analyzed. A novel approach of coating was applied in this study on four different types of PTFE membrane laminated different structured waterproof breathable fabrics. The following findings are drawn from the results of the experiment.

Hydrostatic resistance values of prepared two-layered and three-layered PU membrane laminated fabrics are obtained more than 500 cmH_2O which indicates the fabrics as good quality products for using as outdoor sports clothing. Compactness of outer woven layers with different warp and weft cover factors make the laminated fabrics with different fabric weights and densities which influence on hydrostatic resistance values. From P-value, r-value and R^2-value, it has been found that hydrostatic resistance property is significantly and positively influenced by fabric weight and fabric density. Added knitted layers to three-layered samples contribute to increase fabric weight and density. And these three-layered fabrics show better hydrostatic resistance values than two-layered samples. Sample prepared by outer woven layer with more warp cover factor and weft cover factor or more compact structure shows higher hydrostatic resistance property than others.

Obtained R_{et} values of all prepared PU membrane laminated samples are between 6-10 m^2Pa/W. This means that all prepared samples are good breathable fabrics. Water vapour permeability of the prepared sample fabrics are greatly influenced by fabric weight and fabric thickness. Obtained P-value, r-value and R^2-value from statistical analysis express that water vapour permeability of the samples are significantly and negatively influenced by their fabric weight and fabric thickness. This indicates that the sample fabrics become more comfortable when their fabric weight and thickness are lower. But, fabric weight has more influence than fabric thickness on water vapour permeability of the samples that is derived from statistical analysis. Three-layered samples show less water vapour permeability than two-layered samples due to their increasing weight and thickness after adding knitted inner parts.

Air permeability values of the prepared sample fabrics depend on fabric thickness, fabric density, warp and weft cover factors or compactness of outer woven layers as well as inner knitted structures. Obtained P-value, r-value and R^2-value express significant negative influences of fabric thickness and fabric density on their air permeability values. Prepared sample fabric with lower thickness and lower density is found as more air permeable and sample fabric of more compactness with more warp cover factor and more weft cover factor of outer woven layer is found as less air permeable. Three-layered samples are less air permeable than two-layered samples as knitted inner layer for each three-layered sample causes more air entrapment resulting in less air permeability. However, thermal resistance values of PU membrane laminated fabrics are significantly and positively influenced by their thickness property that has been proved by obtained P-value, r-value and R^2-value from statistical analysis. Three-layered samples show more thermal resistance values than two-layered samples due to their more thickness values after adding knitted parts as inner layers. Moreover, due to using the knitted fabrics, there are more air gaps between fibers that cause the decrease of heat transfer with increasing thermal resistance property. So, prepared three-layered fabrics are more comfortable than two-layered fabrics in comparatively cold condition.

From obtained P-value, r-value and R^2-value, it is evident that breaking strength of the sample fabric is significantly and positively influenced by fabric weight and fabric density. All three-layered samples show more breaking strength property than all two-layered samples due to their higher fabric weight and density after adding knitted fabrics as inner layers. Prepared laminated fabric with outer woven layer of more warp cover factor and more weft cover factor shows higher breaking strength due to the more compactness of the fabric. Again, obtained P-value, r-value and R^2-value determine that bending rigidity of PU membrane laminated fabric is significantly and positively influenced by the thickness property of the fabric. Three-layered sample fabric with outer woven layer of highest warp cover factor and weft cover factor shows the highest bending rigidity value among all the samples due to its highest thickness value with maximum compactness.

During application of coating solution on four different types of PTFE membrane laminated three-layered waterproof breathable sample fabrics, the best mixing ratio of 50 g/L C_6-based fluorocarbon water repellent chemical and 15 g/L polysiloxane hydrophobic softening agent was selected and applied according to the best results obtained from coating a particular sample. However, all coated four samples show more regular and smooth surfaces under scanning electron microscope due to evenly deposition of coating materials on the fabric layers. All the four coated sample fabrics show complete non-wetting property during spray test even after washing which indicates the proper water-repellent property of the four samples after coating. Hydrostatic resistance and breaking strength properties of all four samples are significantly increased after coating as P-values are obtained less than 0.05 as well as F-ratios are higher than F-critical values from ANOVA for both cases during comparison of every coated and uncoated sample fabric. Bending rigidity is also increased after coating, but it is not as much higher increase as the increases of hydrostatic resistance and breaking strength due to using of softening agent. No significant increases or no big differences are obtained in water vapour permeability after coating in four different samples which indicates no significant changes in breathability after coating. And, air permeability is also not increased significantly after coating for any of four sample fabrics.

All the above findings of the experiment should be considered with a great importance during designing and preparing outdoor sports waterproof breathable laminated fabrics for the comfortability of the users.

SCOPE FOR FUTURE WORK

The experimental ideas of this research work have endeavored to introduce the knowledge that could be useful to define the future direction and provide insightful references to researchers. Due to scope of the work, followings are recommended for future work:

- Preparation of the laminated layered waterproof breathable fabrics using outer layer parts with fabrics of different types of fibers and analyzing their comparative studies.
- Preparation of the laminated waterproof breathable layered fabrics using inner layers with lining materials of different types of fibers and analyzing their properties.
- Preparation of the laminated layered fabrics with same outer and inner layers, but different types of membranes, i.e., microporous membrane or hydrophilic membrane with different weight and thickness and studies of their properties in order to use as protective or sports clothing.

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1**. Razzaque, A.** and Tesinova, P. "Analysis of water resistance and mechanical performance of microporous polyurethane membrane laminated waterproof breathable fabrics", paper accepted in TechConnect World Innovation Conference to be held in June, 2019 at Boston, Massachusetts, U.S.A.

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